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DEVELOPMENT OF NEW TEST TECHNIQUES FOR DETERMINING THE COMPATIBILITY OF MATERIALS WITH LIQUID OXYGEN UNDER IMPACT

W. R. Blackstone
B. B. Baber
P. M. Ku

Southwest Research Institute

TECHNICAL REPORT AFAPL-TR-67-41

December 1967



Air Force Aero Propulsion Laboratory
Directorate of Laboratories
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

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AFAPL-TR-67-41

**DEVELOPMENT OF NEW TEST TECHNIQUES FOR
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**W. R. Blackstone
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P. M. Ku**

Southwest Research Institute

FOREWORD

This report was prepared by Southwest Research Institute, 8500 Culebra Road, San Antonio, Texas, under Contract AF 33(615)-2384. The contract was initiated under Project No. 3044, Task No. 304401. The work was administered by the Fuels, Lubrication, and Hazards Branch, Air Force Aero Propulsion Laboratory, Directorate of Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The project engineers were Messrs. G. A. Beane, L. J. DeBrohun, and H. A. Smith (APFL).

This report covers one phase of work performed under the subject contract in the period of February 1, 1965 through December 31, 1966. This report was submitted by the authors February 1967.

This technical report has been reviewed and is approved.

A. V. Churchill
A. V. CHURCHILL

Chief, Fuels, Lubrication, and
Hazards Branch
Support Technology Division
Air Force Aero Propulsion Laboratory

ABSTRACT

This report summarizes the work done toward the development of suitable test methods for evaluating the compatibility of materials with liquid oxygen under impact. The work indicates that the potential explosive hazard of a material may be defined by two parameters: impact sensitivity and reaction intensity. Impact sensitivity is measured in terms of the drop height at which the material has a 50-percent probability of reaction. Reaction intensity is measured in terms of the maximum peak pressure of the air shock wave generated by the reaction. Detailed test methods for determining impact sensitivity and reaction intensity are proposed. It is recommended that tests be run on a wide variety of liquids, greases, and solids, in strict accordance with the proposed test methods, in order to establish their repeatability and reproducibility.

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SECTION I

INTRODUCTION

1. GENERAL

This report summarizes a portion of the work performed at Southwest Research Institute in the period of February 1, 1965 through December 31, 1966, under Contract AF 33(615)-2384, entitled "Lubrication Research for Aero Propulsion Systems." The objectives of this broad program are the development and refinement of lubricant evaluation techniques for aero propulsion systems, and the evaluation of candidate lubricants and lubrication techniques. This report is concerned with only one phase of the overall effort: the development of suitable test methods for determining the compatibility of materials with liquid oxygen under impact. Work performed under other phases has been dealt with in five previous reports^{(1, 2, 3, 4, 5)*} and will be presented in other future reports.

Many organic and inorganic materials that are compatible with liquid oxygen under static conditions may cause explosions in the oxidizer system of a rocket engine when subjected to mechanical impacts. Therefore, the selection of materials to be used, such as lubricants, sealants, gaskets, and thread compounds, must be based on an evaluation of their compatibility with liquid oxygen under the conditions of impact. In the work described herein, an impact tester originally devised by the Army Ballistic Missile Agency† (hereafter referred to as ABMA) for evaluating the compatibility of materials with liquid oxygen⁽⁶⁾ was chosen for further development. The basic ABMA design was selected because it appeared to be more fully developed and more widely used than others.

2. BACKGROUND OF DEVELOPMENT

The ABMA-type impact tester has undergone considerable refinements and standardization over the years, and these efforts have resulted in the adoption of standard impact sensitivity test methods in the forms of USAF Specification Bulletin 527⁽⁷⁾, NASA Specification MSFC-SPEC-106B⁽⁸⁾, and ASTM Tentative Test Method D2512-T⁽⁹⁾. All of these test methods employ the same basic drop-weight type tester with only minor variations in test procedures and in the details of handling the test materials. In all instances, the test material is placed in an aluminum specimen cup in the presence of liquid oxygen, and upon the test material is placed a stainless steel striker

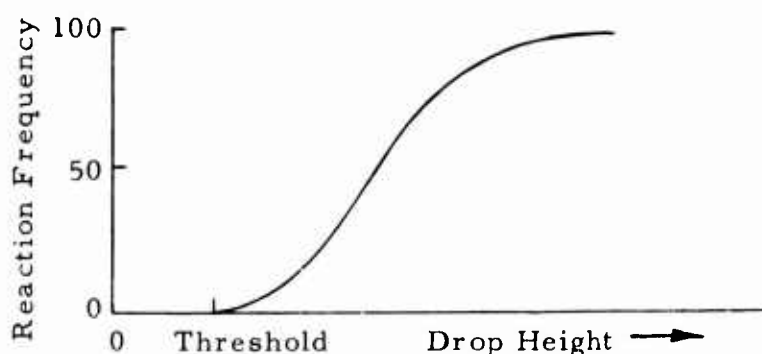
*Superscript numbers in parentheses refer to the List of References.

†Now Marshall Space Flight Center, National Aeronautics and Space Administration.

pin. A plummet is released from a known height, and observations are made as to whether a reaction has or has not taken place. In all three methods, twenty drops each are made from systematically varying heights to obtain the highest drop height that will give no reactions. The drop height so determined is known as the "threshold value" of the material under test. In the Air Force and NASA methods, a variation of the threshold value technique is widely used to qualify materials for service. In this variation, twenty or more drops are made from a fixed height of 42 in. (or 43.3 in.), and the materials are accepted or rejected on a "go or no-go" basis. Clearly, for a material that yields no reactions in this test, its threshold value is, by definition, either 42 in. (or 43.3 in.) or higher.

The principal difficulty with all of these test methods is the poor repeatability and reproducibility of the threshold values obtained. This difficulty was brought out by the results of a cooperative test program conducted by the Air Force⁽¹⁰⁾ and the more recent study made by ASTM⁽⁹⁾. For example, according to the ASTM analysis, for a threshold value of 24 in., the repeatability (one operator and apparatus) is 17.0 in., and the reproducibility (different operators and apparatuses) is 40.8 inches. It must be clear from these figures that the test methods, as they now stand, are virtually incapable of discriminating the impact sensitivity of materials with any confidence.

The cause of this poor precision has generally been thought to lie in the variations in equipment or procedure. Early in this program, however, it became clear that the main trouble arose from the statistical inadequacy of the "threshold value" method of test. The weakness of this concept may be seen by considering the probability distributions one is obliged to work with in any type of sensitivity experiment⁽¹²⁾. In testing the impact sensitivity of, say, an explosive, one cannot measure the precise energy input level (threshold value) which would barely initiate a reaction but can only observe whether an applied drop height does, or does not, initiate a reaction in a given sample. The sample used is consumed or changed as a result of the trial; therefore, a new sample must be prepared for the next trial. It is highly improbable that the original sample can be exactly duplicated; therefore, the new sample will most probably have a different threshold value from the original. Thus, in a whole population of samples, there would be some sort of probability distribution of threshold values with the result that, in a test of a number of samples impacted from a given height, some will react and other will not. A plot of the frequency of reactions over a range of drop height yields a cumulative probability distribution which, with most explosives, has been found to exhibit the following trend:



It so happens that the threshold value determination involves the determination of the end point of the lower tail of the probability curve--a point which is statistically most difficult to ascertain even with a very large number of test drops, not to mention a relatively small number such as ordinarily required in a practical test method. In other words, the evaluation of impact sensitivity in terms of threshold values, however psychologically desirable it may be, is inherently at a statistical disadvantage and cannot assure reasonable precision from a relatively small number of test drops.

In order to utilize a limited number of test drops efficiently in determining impact sensitivity, it is clear that the test drops should be made in the neighborhood of the midpoint of the probability curve. In this region, the occurrences are frequent, and the relation between reaction frequency and drop height is, with most explosives, nearly linear if the drop height is expressed in logarithmic scale. By concentrating all of the test drops in this region in such a way as to bracket the midpoint of the probability curve, then the information from every test drop is utilized in the search for the midpoint, and, therefore, the midpoint is determined with the greatest possible statistical reliance. This procedure is explained in Reference 12. A step-by-step description of an impact sensitivity test method, comprising twenty-five test drops in the up-and-down manner, is given in the Appendix of this report. This method determines the "50-percent point" of the material under test, or that drop height which will give a 50-percent probability of reaction for the material.

The up-and-down test had been applied successfully in many other types of material evaluation and thus appeared ideally suited to the liquid oxygen impact test. However, before attempting this new test technique, two significant equipment changes were made to eliminate suspected sources of variation. One of these suspected sources was the plastic deformation of aluminum specimen cups under impact.⁽¹¹⁾ It was found that the aluminum cups experienced measurable permanent deformations at drop heights of 12 in. and much more at higher drop heights. Further, the deformation was not repeatable at any given drop height to such an extent that the rebound height of the plummet varied greatly from drop to drop. Accordingly, the impact stimulus experienced by the test material must also vary greatly from drop to drop, because of the varying energy absorption of the aluminum in deformation.

In order to minimize the variability of specimen cup deformation, experiments were made using stainless steel as the cup material in place of aluminum⁽¹¹⁾. It was found that, when stainless steel specimen cups were used in conjunction with stainless steel striker pins of 0.7-in. diameter or over, variations in the plummet rebound height became negligible (about 1 percent at 48-in. drop height). In addition, no appreciable permanent deformation of the impact surface of either the cup or the pin was observed.

The other equipment change consisted of installing a rebound catcher⁽¹¹⁾ to prevent secondary impacts by the rebounding plummet. This was done to facilitate the determination of initial impact stimuli by eliminating the complications introduced by secondary impacts.

It was of course known that these equipment changes would probably alter material responses in the impact test. Indeed, such changes were desired because it was also known that probability (reaction frequency) curves constructed from Bulletin 527 data on liquids and greases did not adequately satisfy a basic condition for use of the up-and-down test. This condition is that the probability curve be of appropriate shape, i. e., that the variate (drop height), or some transformation thereof, be reasonably normally distributed. Considering this, it can be seen that the problem now was to determine whether material responses with the new equipment would satisfy this condition. To find the answer to this, an investigation⁽¹¹⁾ was then made using the new stainless steel specimen cups together with both 0.5-in. and 0.7-in. diameter striker pins. The probability curves obtained with the 0.5-in. diameter striker pins deviated from normality in about the same manner as did those obtained from Bulletin 527 data, although the reaction frequencies were somewhat higher. The curves obtained with the 0.7-in. diameter striker pins, however, were much improved, showing a shape not far from log-normal in the middle region.

It appeared from the shape of its probability curves that the 0.7-in. diameter striker pin could be used for the up-and-down test. However, comparison of the curves yielded by the two different diameter striker pins suggested that a further increase in striker pin diameter might yield even more improvement. This indeed proved to be the case, as probability curves for liquids and greases obtained with 0.855-in. diameter striker pins were found to be very close to log-normal in the range of 20 to 80 percent. These curves also showed much improved slopes (a measure of scatter) over those obtained with the 0.7-in. diameter striker pins. With these findings, the basic distribution requirements of the up-and-down test were satisfied, and advantage could be taken of this method's improved precision.

The up-and-down test method was then applied to the determination of the 50-percent points of a number of liquids and greases ranging from common hydrocarbons to highly fluorinated materials (Section II. 2). Stainless steel specimen cups and 0.855-in. diameter stainless steel striker pins were used

in this test series. During the test series, the repeatability of the 50-percent determinations was checked through numerous replicate tests and was found to be excellent; for example, individual 25-drop tests on a grease yielded 50-percent points deviating no more than 1.3 in. from the average of 10 such tests. The results of this test series showed the startling fact that, with improved test repeatability, the impact sensitivities (50-percent points) of most greases and liquids actually did not vary a great deal (about 5 to 16 in. for greases and 11 to 20 in. for liquids). Furthermore, within these already close groupings of liquids and greases, the sensitivities of several materials known to present little or no hazard in contact with LOX (such as chlorofluorocarbon oils and greases) were practically the same as those of other very hazardous materials (such as ordinary silicone oils or greases).

The above results raised considerable doubt as to whether impact sensitivity was indeed the parameter best describing relative compatibility. The main reason for this doubt was the behavior of some "relatively inert" materials, which showed evidence of reaction at about the same stimulus levels as the highly explosive materials, yet their reactions were always very faint--in fact, almost undetectable. It was obvious that, from a practical standpoint, these materials presented little or no fire or explosion hazard and that they were thus much more compatible than the explosive materials; yet, it was also obvious that such behavior was not being meaningfully described by sensitivity measurements. The reason for this also became clear: a sensitivity measurement, by definition, is concerned only with the stimulus required to initiate a reaction, and can give no information whatever about what happens to the reaction after it starts. From these various considerations, it was concluded that the parameter best describing the compatibility of liquids and greases would be the intensity of reaction after initiation. This conclusion led to the development of a method for obtaining quantitative measurements of reaction intensity (Sections II and III).

The technique worked out for measuring reaction intensity involves the measurement of the peak pressure of the air shock wave generated by an impact-induced reaction. As described in the reaction intensity test method outlined in the Appendix of this report, these measurements are made on each of twenty test drops from a fixed height of 43.3 inches. Using this test on a wide variety of liquids and greases, it was found that all the materials tested could easily be classified as either relatively inert or explosively reactive. Extensive tests demonstrated that the method was practical and highly repeatable. Further, it was shown that the compatibility differences between the relatively inert and the more reactive liquids and greases, which could not be shown with sensitivity measurements, became remarkably apparent when viewed in terms of peak pressure measurements. Thus, it became clear that, for liquids and greases, reaction intensity alone could provide a complete description of the fire or explosion hazard (Section IV.4).

Other compatibility questions of considerable concern in the past have involved such factors as the effect of trace contaminants on the reactivity of a material, and what is known as batch-to-batch variance of sensitivity. Using the reaction intensity test, it was found that small amounts of even a highly explosive contaminant did not cause a "relatively inert" material to become explosive. This suggested that it would be difficult to blame an "unexplained" reaction of some material on the presence of trace contaminants. Further, considering both the reaction intensity information and the up-and-down test sensitivity data, no evidence could be found that there was any such thing as batch-to-batch variance of sensitivity. It appears that all batches of such materials are potentially hazardous, with their apparent batch-to-batch variance being attributable primarily to the variance inherent in the current test methods.

It was found that solid materials behaved quite differently from liquids and greases, in that sensitivity proved to be a significant parameter for evaluating compatibility (Section IV.3). However, reaction intensity still appeared to be a valid adjunct, since some solids gave low-order reactions while others gave more violent reactions.

3. TEST MATERIALS

The sample materials referred to in this report are identified in Table 1.

TABLE 1. IDENTIFICATION OF TEST SAMPLES USED IN
COMPATIBILITY TEST PROGRAM

Sample Code	Sample Type	Description	Manufacturer
D-1071	Liquid	Fluorochemical FC 43	Minnesota Mining & Mfg. Co.
D-1093	Liquid	DC 510	Dow Corning Corp.
E-1005	Liquid	FS-1265	Dow Corning Corp.
E-1009	Liquid	Aroclor 1254	Monsanto Chemical Corp.
E-1010	Liquid	Halocarbon 411 V	Halocarbon Products Corp.
F-1018	Grease	Molykote G	Alpha-Molykote Corp.
F-1040	Liquid	FX 45	Minnesota Mining & Mfg. Co.
G-1051	Grease	Supermil M-125	American Oil Co.
G-1052	Grease	MCG-63-46	(a) (b)
G-1071	Grease	MLO-62-1009B	(a) (b)
G-1074	Liquid	QF-6-7012	Dow Corning Corp.
G-1077	Grease	EC-2388, graphite in fluoro-carbon oil	Acheson Colloids Co.
H-1012	Grease	EC-2292, MoS ₂ in fluoro-carbon oil	Acheson Colloids Co.
H-1013	Grease	EC-2450, tetrafluoroethylene in fluorocarbon oil	Acheson Colloids Co.
H-1027	Grease	Different lot of G-1051	
H-1032	Grease	KEL-F 90	Minnesota Mining & Mfg. Co.
H-1040	Grease	LOX-SAFE	Redel, Inc.
H-1081	Grease	FS-1281	Dow Corning Corp.
H-1085	Grease	Different lot of H-1032	Minnesota Mining & Mfg. Co.
J-1012	Grease	EC-1730	Acheson Colloids Co.
J-1013 thru J-1019	Grease	Different lots of H-1040	
J-1022	Grease	DC-4 compound	Dow Corning Corp.
J-1050	Liquid	MIL-L-6082, Grade 1100	Humble Oil and Refining Co.
J-1052	Liquid	KX 295	Minnesota Mining & Mfg. Co.
J-1053	Grease	Lubriplate No. 630-AA	Fiske Brothers Refining Co.
J-1074	Grease	Different lot of H-1032	Minnesota Mining & Mfg. Co.
J-1080	Liquid	Ucon LB-65	Union Carbide Chemicals Co.
J-1120	Grease	KX-91	Minnesota Mining & Mfg. Co.
K-1001	Grease	FS-1292 (MIL-G-27617), Lot 0620	Dow Corning Corp.
K-1002	Grease	Different lot of H-1032	
K-1015	Grease	MCG-65-51	(a)
K-1022	Grease	Braycote 617	Bray Oil Co.
K-1081	Liquid	Different lot of J-1080	
K-1082	Liquid	Different lot of E-1010	
None	Solid	Acetate (clear)	Cellanese Corp.
None	Solid	Phenolic (canvas base)	St. Regis Paper Co.
None	Solid	Mylar	E. I. duPont deNemours & Co.
None	Solid	Kapton	E. I. duPont deNemours & Co.
None	Solid	Teflon	E. I. duPont deNemours & Co.
None	Solid	Vinyl	Bakelite Corp.

(a) Received through AFAPL from AFML (MANL).

(b) Different batches of the same nominal formulation.

SECTION II

DEVELOPMENT OF TEST APPARATUS AND PROCEDURES

1. TEST APPARATUS

The work reported herein was carried out on an ABMA impact tester, with certain modifications incorporated as a result of investigations conducted at SwRI. A complete description of the basic ABMA impact tester may be found in several standard references^(7, 8, 9) and therefore will not be repeated here.

The modifications made by SwRI included principally the introduction of an all-stainless-steel anvil region assembly and the addition of a reaction intensity measuring system. One version of the all-stainless-steel anvil region assembly, which will be referred to as Anvil Region Assembly III for convenience, was developed prior to the current reporting period and was discussed in detail in a previous summary report⁽¹¹⁾. Briefly, Figure 1 shows the stainless steel striker pin. Figure 2 shows the stainless steel specimen cup with a Teflon sleeve on top for containing liquid oxygen and another Teflon sleeve at bottom for centering the cup in the cup holder. Note that the specimen cup design dictates a test sample thickness of 0.020 in. for liquids and greases. Figure 3 shows the entire Anvil Region Assembly III.

The reaction intensity measuring system was developed during the current reporting period. The design of this system, along with its history of development, will be dealt with in appropriate sections of this report.

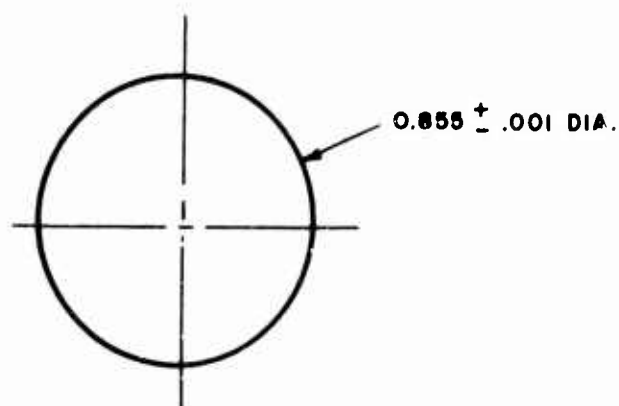
In addition to the above, a rebound catcher was installed on the tester. Use of this catcher precluded secondary impacts on the test samples in all of the tests related herein.

2. IMPACT SENSITIVITY MEASUREMENTS

The impact sensitivity of a material is expressed, in this report, in terms of the drop height at which the material gives a 50-percent probability of reaction with liquid oxygen. This drop height for 50-percent probability of reaction is called, for brevity, the "50-percent point." The 50-percent point is determined by means of an up-and-down test procedure first experimented with in the previous reporting period⁽¹¹⁾, finalized in the current reporting period, and outlined in its final form in the Appendix of this report. It involves, in effect, twenty-five test drops from varying heights in such a manner as to bracket the probable 50-percent point of the material under test. For this reason, the test drops are used in the most effective fashion statistically; hence, the 50-percent point is statistically superior to the "threshold value" currently used in the standard test methods.

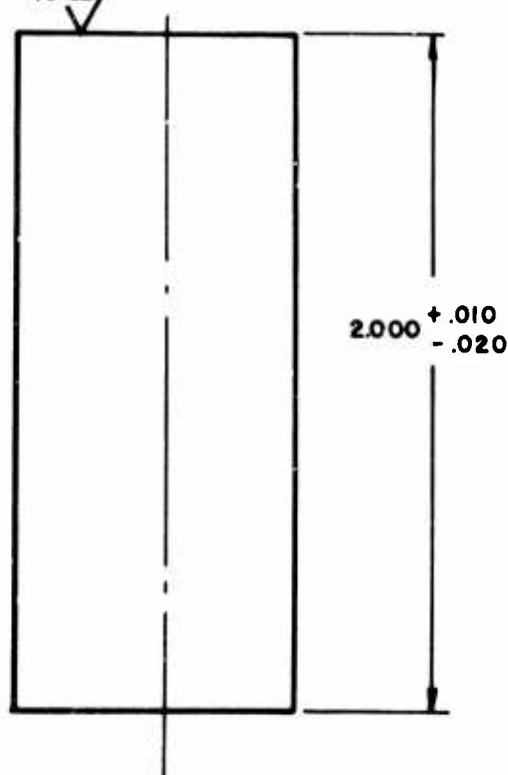
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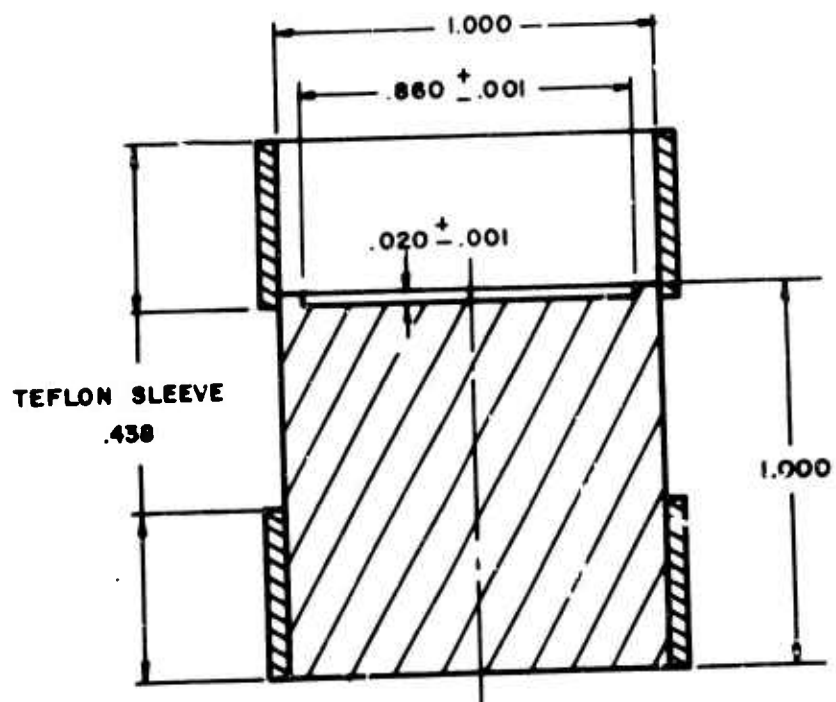
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MATERIAL:
17-4 PH STAINLESS STEEL
HEAT TREAT:
CONDITION H-900

92821 K

FIGURE 1. STAINLESS STEEL STRIKER PIN



MATERIAL:
 17-4PH STAINLESS STEEL
 HEAT TREAT:
 CONDITION H-900

92825 K

FIGURE 2. STAINLESS STEEL SPECIMEN
 CUP WITH TEFLON SLEEVES

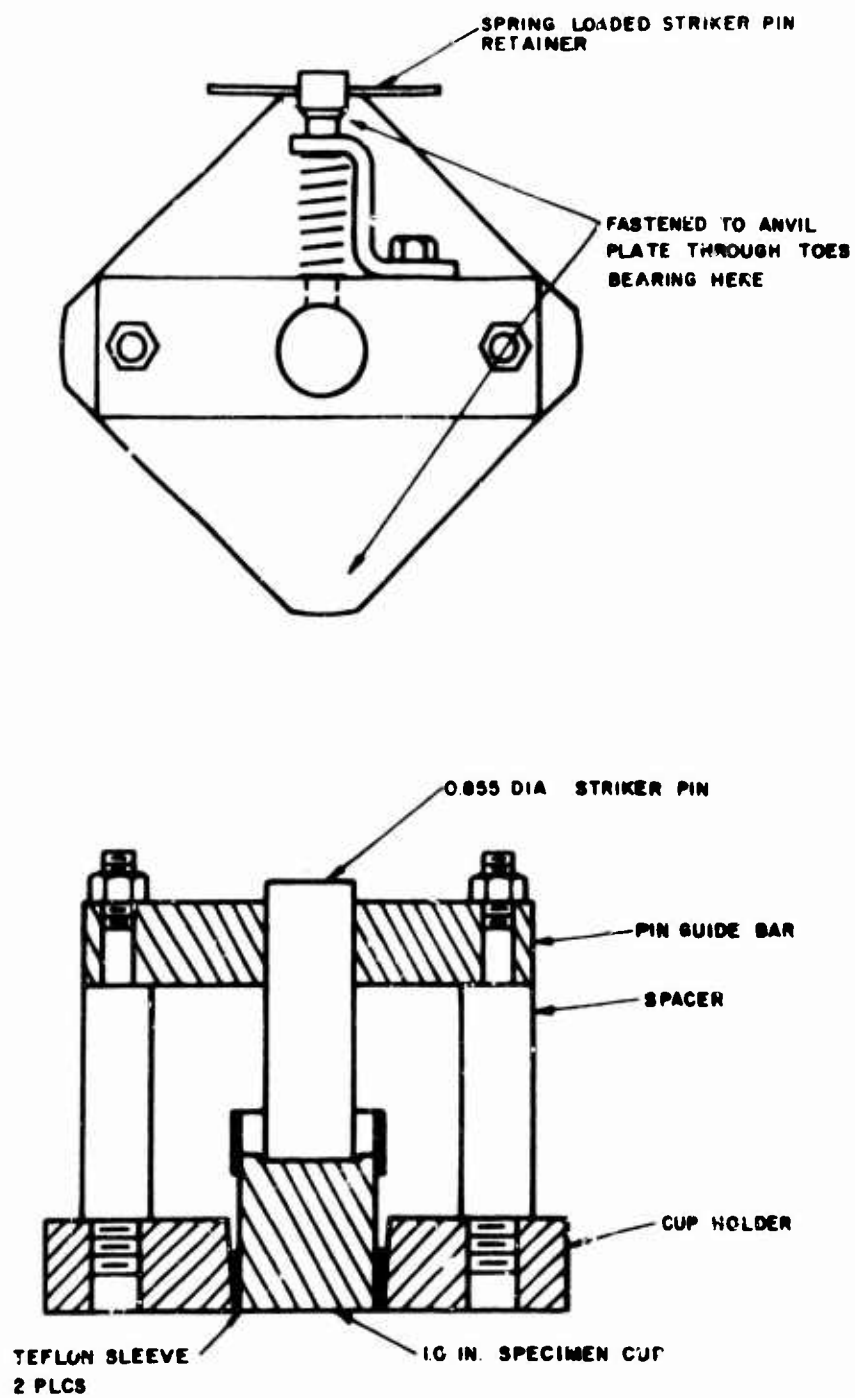


FIGURE 3. ANVIL REGION ASSEMBLY III

The 50-percent points of a number of greases were determined in the previous reporting period, using Anvil Region Assembly III. These data are reproduced herein in Table 2 and Figure 4 in order to provide the basis for comparison with the data on liquids and solids obtained during the current reporting period. Table 2 shows the repeatability of the test on grease J-1012, based on the results of ten replicate tests. Figure 4 compares the 50-percent points of the various greases evaluated. For greases tested two or more times, the highest and lowest ratings are shown in the figure. The letter "P" under the sample code indicates that the sample has passed the USAF Specification Bulletin 527 test; the letter "F" denotes that the sample has failed the Bulletin 527 test.

The repeatability of the test is generally better for greases than for liquids. Table 3 shows the results of ten replicate tests on G-1074, a liquid. Note that the standard deviation (logarithmic scale) in this case was almost twice that for grease J-1012 (Table 2).

Figure 5 shows the relative impact sensitivities of several liquids as determined by the up-and-down test method. For G-1074, the highest and lowest determinations from Table 3 are shown. As in the graph for greases, the letter "P" under the sample code indicates that the sample has passed the Bulletin 527 test, and the letter "F" denotes that the sample has failed the Bulletin 527 test.

The repeatability of the test is generally better for solids than for greases. This is illustrated in Table 4, showing the results of ten replicate tests on solid acetate. Although the standard deviation (logarithmic scale) in this case was only half of that for grease J-1012, the determinations as such were probably not entirely accurate. This is because the 50-percent point of the acetate is so close to the upper drop height limit of the ABMA tester that no reactions were noted at 48.0 in. on several occasions. In such cases, the up-and-down test method requires the next drop to be made from a greater height which, of course, was not possible. Therefore, when this situation occurred, the next drop was made from the same height. The consequences of such a departure from the test method are not known; however, it is logical to presume that the determination of the 50-percent point would be influenced at least slightly. Fortunately, this situation has not been observed with the rather large number of greases and liquids evaluated so far, nor with many other solids evaluated (Sections II.6 and IV.3). In the case of solid acetate, it was found that the use of striker pins of smaller diameter had the effect of reducing the 50-percent point and therefore bringing the test to within the limit of the tester. (11)

Only two more solids were rated at this early developmental stage. For phenolic, a 50-percent point of 1.436 (log scale) or 27.3 in. was obtained. Mylar was not on hand in the 0.020-in. sample thickness used in the other tests; therefore, a thickness of 0.005 in. was used, producing a

TABLE 2. REPEATABILITY OF 50-PERCENT
POINTS FOR GREASE J-1012

<u>Test No. †</u>	<u>Logarithm of 50-Percent Point</u>	<u>50-Percent Point, * in.</u>
1	1.010	10.2
2	0.987	9.7
3	0.936	8.6
4	0.943	8.8
5	1.034	10.8
6	1.021	10.5
7	0.989	9.8
8	1.031	10.7
9	1.002	10.0
10	1.038	10.9

*Average 50-Percent Point = 0.999 (log scale) = 9.9 in.; Standard Deviation = 0.0360 (log scale).

†Each test consisted of twenty-five drops made by the up-and-down method, using Assembly III.

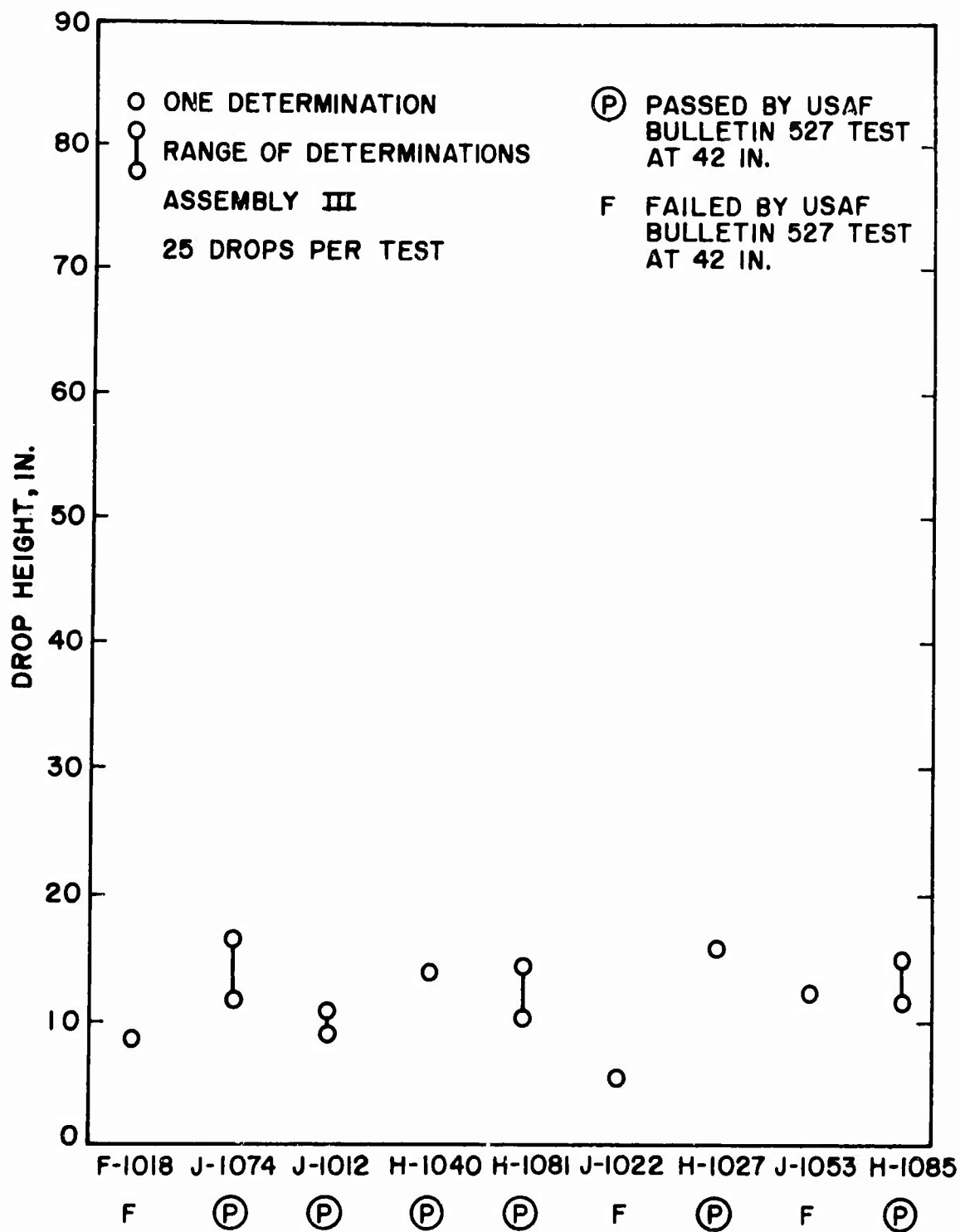


FIGURE 4. RELATIVE IMPACT SENSITIVITIES OF VARIOUS GREASES

TABLE 3. REPEATABILITY OF 50-PERCENT
POINTS FOR LIQUID G-1074

<u>Test No. †</u>	<u>Logarithm of 50-Percent Point</u>	<u>50-Percent Point, * in.</u>
1	1.134	13.6
2	1.131	13.5
3	1.116	13.0
4	1.108	12.8
5	1.179	15.1
6	1.193	15.6
7	1.049	11.2
8	1.239	17.3
9	1.223	16.7
10	1.265	18.4

*Average 50-Percent Point = 1.164 (log scale) = 14.6 in. ; Standard Deviation = 0.0675 (log scale).

†Each test consisted of twenty-five drops made by the up-and-down method, using Assembly III.

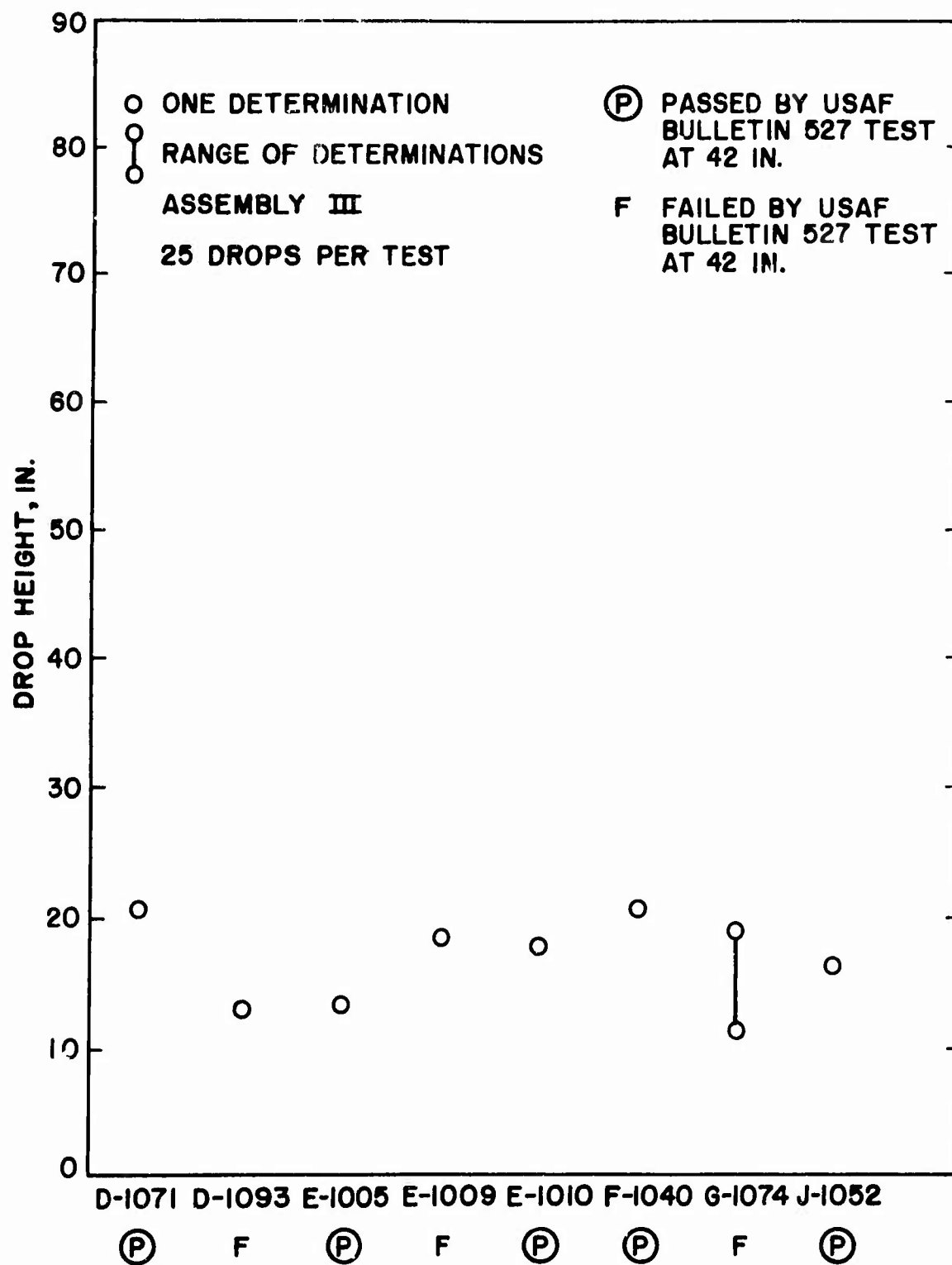


FIGURE 5. RELATIVE IMPACT SENSITIVITIES OF VARIOUS LIQUIDS

TABLE 4. REPEATABILITY OF 50-PERCENT
POINTS FOR SOLID ACETATE

<u>Test No. †</u>	<u>Logarithm of 50-Percent Point</u>	<u>50-Percent Point, * in.</u>
1	1.597‡	39.5
2	1.610	40.7
3	1.617‡	41.4
4	1.619	41.6
5	1.631	42.8
6	1.648‡	44.4
7	1.610‡	40.7
8	1.610	40.7
9	1.633	43.0
10	1.660‡	45.7

*Average 50-Percent Point = 1.624 (log scale) = 42.1 in.; Standard Deviation = 0.0183 (log scale).

†Each test consisted of twenty-five drops made by the up-and-down method, using Assembly III.

‡This determination is somewhat questionable because the occurrence of no reaction at the highest obtainable drop height necessitated a departure from the requirements of the test procedure, as discussed in the text.

50-percent point of 1.304 (log scale) or 20.1 inches. Because of the differences in thickness and the small number of solids tested, no graph showing the relative impact sensitivities of solids is included at this stage.

At this point, it should be remarked that the most statistically reliable test method available was used in determining the impact sensitivity ratings of the materials. Considering Figures 4 and 5, it will be noted that the liquids as a group were slightly less sensitive than the greases. Also, taking the highest and lowest determinations for G-1074 as an indication of the test-to-test variance, it could be argued that all the liquids tested actually had very nearly the same sensitivity (11 to 20 in.). For the greases, all the sensitivities fell between about 5 to 16 inches. Considering that the liquids and greases tested ranged from common hydrocarbons to highly fluorinated materials presently used in LOX systems and thus should cover the range of prime interest, it becomes apparent that impact sensitivity as such does not provide a meaningful measure of the degree of hazard of such materials. Yet, there are obvious differences in the performance of the various materials that did not show up in the impact sensitivity test method, at least in a quantitative sense. These are the tremendous differences in the violence of the reactions. It was these considerations that led to the investigation of reaction intensity, which will now be described.

3. FEASIBILITY OF REACTION INTENSITY MEASUREMENTS

In the course of the previously discussed 50-percent point tests on various liquids, greases, and solids, hundreds of reactions were obtained. During these tests, it was observed that audible and visible differences in the intensities of the reactions obtained were quite pronounced. Some materials tended to explode violently, while others gave such faint reactions that it was sometimes difficult to decide whether or not a reaction had in fact occurred. It was also noted that the general intensity level associated with many materials seemed to remain relatively constant. For example, the reactions obtained with H-1074 were always very faint while those obtained with G-1074 were nearly always quite violent. In view of these observations and the rather inconclusive separation of the impact sensitivity ratings, it was decided that the feasibility of measuring reaction intensities should be investigated.

It would, of course, be preferable to obtain a direct, quantitative measurement of either the pressure or the temperature associated with a reaction; however, such a measurement did not appear to be feasible with the configuration of the basic test apparatus. On the other hand, a relative measurement of either pressure or temperature made just outside the reaction zone would be adequate for the purpose of comparing reaction intensities if the differences were significantly pronounced. Since this seemed to be the case from the observations previously noted, it was decided that the detection method most readily applicable to the present impact test apparatus would be the measurement of the pressure peak in the air shock wave generated by a reaction.

To obtain an idea of the type of shock pressures being dealt with, a quartz piezoelectric pressure transducer (Kistler Model 601) was mounted at a point 6 in. from the nearest edge of the specimen cup. The axis of the transducer was placed normal to the line of sight to the specimen cup to avoid both the measurement of stagnation pressure and damage to the transducer diaphragm by flying particles of the Teflon sleeve or sample material. The transducer signal was fed through a Kistler Model 566 electrostatic charge amplifier to a Type CA plug-in unit in a Tektronix Model 533A oscilloscope. Contact of the falling plummet with a microswitch provided the triggering pulse for the single sweep of the oscilloscope trace.

Figure 6 shows three typical shock wave traces for grease J-1053 impacted from a drop height of 30 inches. The abscissa represents time reading from right to left, and the ordinate represents pressure. The ordinate and pressure scales are shown on the figure. The magnitude of peak pressure in each case is shown under each trace. It will be noted that, in addition to the primary pressure peak, there are also a number of secondary peaks. These secondary peaks were attributed in part to reflections of the shock wave from the plummet guide rails. Such reflected waves could, and probably did, strike the transducer at other than a grazing incidence, thus producing stagnation pressures. In addition, Teflon sleeve particles could have struck the transducer or its cable, producing acceleration signals. This situation was considered undesirable; therefore, it was decided to try to reduce the influence of the sources of the secondary peaks.

Figure 7 shows a sketch of the device used to improve the signal-to-noise ratio. The shock wave probe is a stainless steel tube 16 in. long and 1.625 in. in diameter. A shock wave traveling through the tube cannot expand; thus the pressure at the outlet end would be the same as that at the inlet end except for relatively small wall losses. In addition, reflected waves which strike the transducer are greatly diminished from expansion and from having traveled longer distances. The transducer, mounted on a heavy ring stand (not shown) and shielded from the reaction area, was placed 0.375 in. from the outlet end of the tube with the diaphragm in a plane tangent to the inside wall of the tube. The inlet end of the tube was 2.375 in. from the nearest edge of the specimen cup. The tube incline was 30° , and the tube axis was pointed at the center of the specimen cup depression. The improvement in signal-to-noise ratio afforded by use of the shock wave probe can be seen in the oscilloscope traces discussed below.

Figures 8 and 9 show the shock wave traces obtained for five other greases and one liquid, also from a drop height of 30 inches. The ordinate and abscissa scales, as well as the magnitude of peak pressure, are shown in each case. It will be noted that grease J-1012 produced two very low order reactions and one moderate reaction. The average intensity of the reactions of J-1012 was thus considerably less than that of the other five greases shown in Figures 8 and 9. Figures 8 and 9 show that the pressure traces were



J-1053
22.4 psi

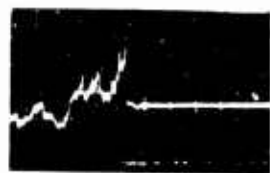
11.2 psi

I



J-1053
25.8 psi

I
200 μ sec



J-1053
29.1 psi

FIGURE 6. SHOCK WAVE TRACES

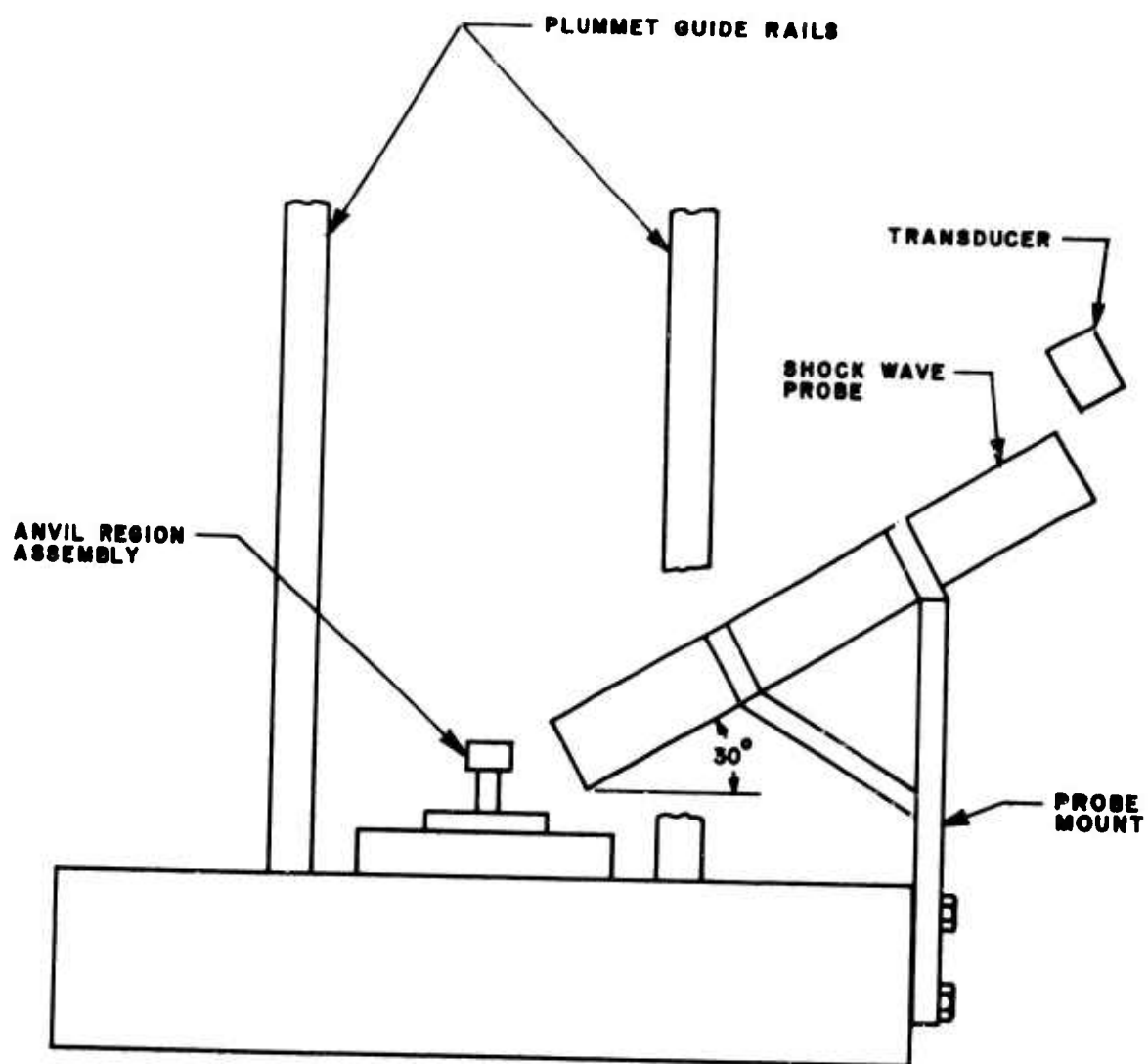


FIGURE 7. SHOCK WAVE PROBE

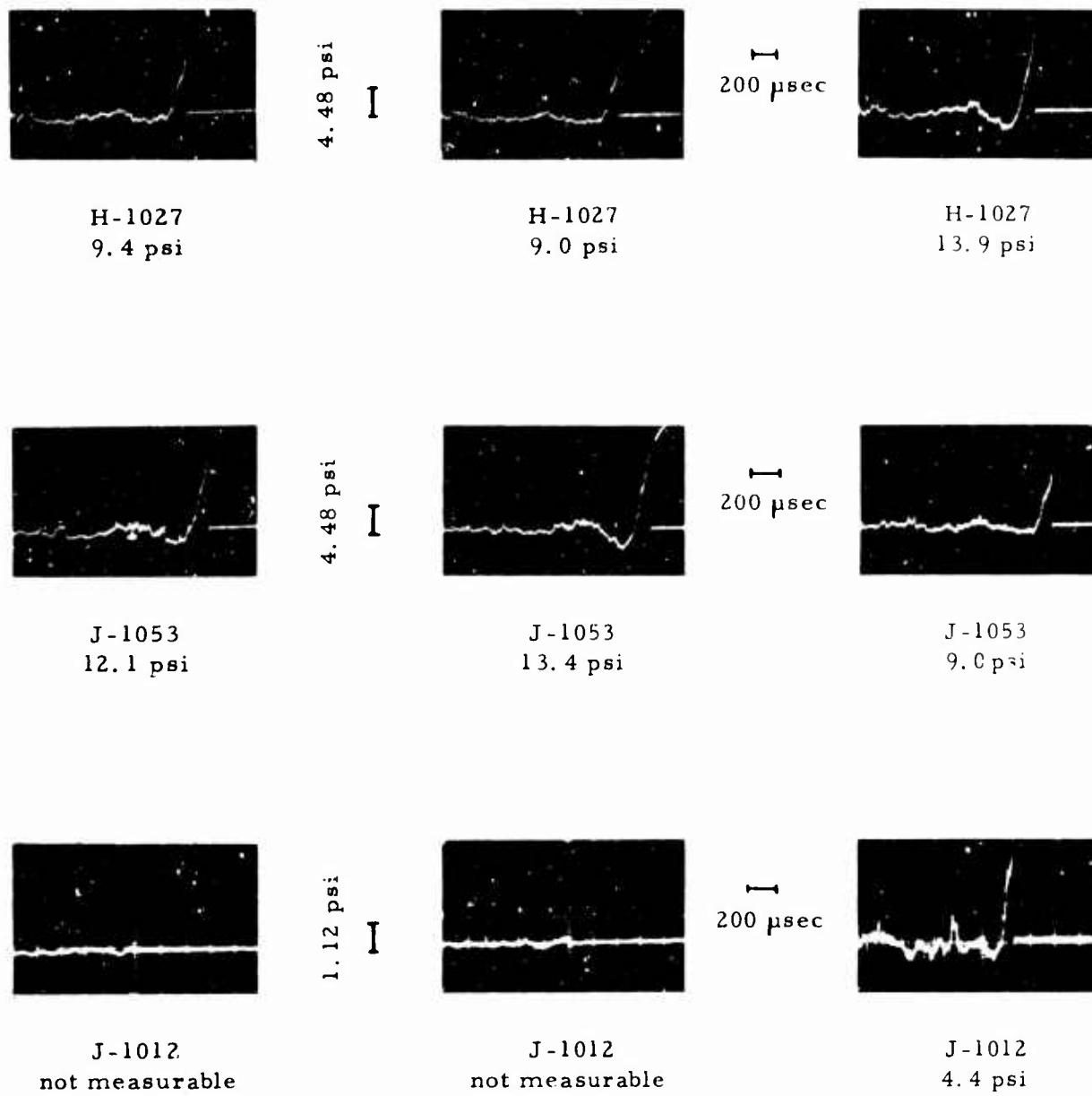
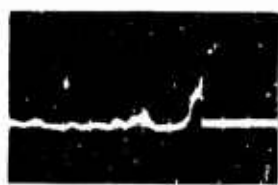
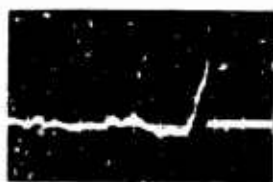


FIGURE 8. SHOCK WAVE TRACES



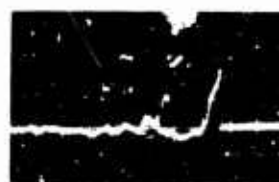
F-101
7.2 psi

4.48 psi



F-1018
10.3 psi

200 μ sec



F-1018
9.4 psi



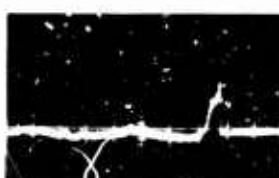
J-1022
10.8 psi

4.48 psi



J-1022
10.8 psi

200 μ sec



J-1022
8.1 psi

4.48 psi



G-1074
17.0 psi

40 μ sec

FIGURE 9. SHOCK WAVE TRACES

generally more free of spurious waves than those presented in Figure 6, due to the use of the shock wave probe. Further, all these figures show that the basic wave form was similar, and the rise time for attainment of peak pressure was of about the same order. Accordingly, it was felt that the magnitude of the peak pressure would provide an adequate description of the reaction intensity for each drop. It is evident from these figures that the peak pressure for ostensibly identical drops could vary considerably, presumably due to the non-repetitive nature of the impact-induced reaction. Therefore, although reaction intensity appeared to be a feasible way of rating materials and appeared to be capable of differentiating violent reactions from faint ones, it was necessary to evaluate the repeatability of the reaction intensity measurements in relation to the intensity level to be distinguished. A large number of repetitive tests would obviously be required to develop this type of information. For such a test program, measurements from the oscilloscope traces did not appear feasible. Therefore, direct measurement of the peak pressure from each drop was considered as the logical alternative.

4. EXPLORATORY MODEL OF A PEAK PRESSURE METER

The above considerations led to the decision to devise a means of reading peak pressures directly on a DC millivoltmeter. To store a rapid transient signal until the voltmeter could respond to it required a special capacitance circuit. No commercial device of the desired capabilities could be found; therefore, one was developed. Essentially, this circuit consists of:

- (1) A fast-response capacitor (1- μ sec rise time) which in turn charges a slow-response capacitor. The two-capacitor system is necessary to combine fast response with slow bleedoff so that the voltmeter will give a steady indication of the peak pressure for a long enough time interval for the operator to take the reading,
- (2) An amplifier to make the output level of the charge amplifier compatible with the voltmeter,
- (3) A diode to prevent the negative portion of the signal from registering the voltmeter, and
- (4) A reset button to discharge the circuit after taking the voltmeter reading.

The peak pressure value was obtained from the voltmeter reading by means of calibration.

5. EXPLORATORY REACTION INTENSITY MEASUREMENTS ON LIQUIDS AND GREASES

Peak pressure measurements were made on a number of liquids and greases. The shock wave probe of Figure 7 was used, but the transducer

was remounted in a support attached to the tester base plate with improved vibration attenuation.

Six series of tests were conducted. The first series involved peak pressure measurements made in conjunction with up-and-down tests of twenty-five drops each. The purpose of this test series was to examine which of the two criteria, impact sensitivity and reaction intensity, would provide a better separation of materials. The second series comprised peak pressure measurements made in tests of twenty drops each from three different drop heights. The purpose of this test series was to gain a quantitative knowledge of the repeatability of the peak pressure measurements under controlled conditions. The third series included peak pressure measurements made in tests of twenty drops each from a given drop height but using three different anvil region assembly and sample thickness combinations. The fourth series comprised peak pressure measurements made in tests of twenty drops each from a given drop height but with the shock wave probe moved closer to the reaction area. The fifth series involved simultaneous measurements, using two independent measuring systems, at locations both equidistant and nonequidistant from the end of the shock wave probe. The purpose of the last three test series was to determine the effects of such variables as specimen cup and striker pin design and materials, sample thickness, and location of the shock wave probe and transducer on the magnitude of peak pressure measurements. Still a sixth test series included twenty drop tests from 43.3 in. using liquid nitrogen in place of liquid oxygen on several materials to see if the reaction intensity measuring technique could aid in distinguishing reactions from nonreactions.

The exploratory model peak pressure meter was employed in this program with the exception of the fifth test series for which an oscilloscope and a camera were used in order to observe the wave forms produced by a new transducer.

In these various series of tests, the peak pressure from each drop was read. For determining sensitivity, reactions were counted in the usual manner on the basis of audible reports, bright flashes, or char marks. In the range of more violent reactions, measurement of peak pressure presented no problem whatever. On the other hand, some reactions were characterized only by char marks, with the resultant air shock waves being too faint to register on the peak pressure meter. In such cases, a positive reaction was recorded for the purpose of determining sensitivity, but the peak pressure was necessarily observed as zero. In the interests of conserving space, the peak pressure readings from the individual drops will not be reported herein. However, the "average peak pressure," computed by averaging the peak pressures from those drops in which reactions had occurred, will be tabulated. In addition, the "maximum peak pressure," which is the maximum value of the peak pressures from those drops in which reactions had occurred, will also be tabulated. From these tabulated values, some idea can be had of the average reaction intensity level and the scatter of the reaction intensity measurement.

a. Up-and-Down Tests

Table 5 presents the results obtained on nine greases using the up-and-down test. Note that, of the greases tested, only F-1018 and J-1022 had distinctly low 50-percent points. There was very little separation among the other seven greases within the limits of repeatability of the 50-percent point.

The separation of grease performance on the basis of peak pressure was, however, much wider. On this basis, F-1018, J-1053, and H-1027 gave violent reactions; J-1022, H-1081, H-1040, and J-1012 gave moderate reactions; and H-1085 and J-1074 gave almost imperceptible reactions. Note that the average peak pressure and the maximum peak pressure generally followed the same trend. Further, although the repeat tests in each case give considerable scatter in peak pressure readings (it should be borne in mind that the drop height had to be varied in an up-and-down test), nevertheless, the separation of the general reaction intensity level of the various greases was wide enough so that the differences noted above seem to be real.

Table 6 summarizes the results on ten liquids using the up-and-down test. This table covers liquids ranging from an aircraft engine mineral oil to highly fluorinated materials--a list which runs the gamut of reactivities. Yet, the separation of the ten liquids on the basis of 50-percent points was not truly distinct. Separation on the basis of reaction intensity was, however, far more pronounced, with J-1050, J-1080, G-1074, and D-1093 being violent reactors; E-1005 being a moderate reactor; and D-1071, F-1040, E-1009, E-1010, and J-1052 being very weak reactors.

As a further check on the ability of the reaction intensity measurement to separate materials, a series of up-and-down tests was run with various mixtures of a relatively inert chlorofluorocarbon oil (E-1010) and a violently reactive polyalkylene glycol (J-1080). Mixtures of these two liquids were tested in an Air Force cooperative test program using the Bulletin 527 method⁽¹⁰⁾. Table 7 shows the results obtained from this test series using the up-and-down test procedure. It is seen that the drop height for 50-percent probability of reactions was virtually the same for all mixtures up to 70 percent of the violent reactor, and the 50-percent point was only slightly lower for the 100-percent violent reactor. On the other hand, the peak pressure measurements show that the reaction intensity was vastly different for the two liquids and that, except for a relatively low percentage of the violent reactor (less than 10 percent), increasing the proportion of the violent reactor increased the reaction intensity progressively and in an accelerated fashion.

b. Effect of Drop Height

Table 8 presents the results obtained on three selected liquids (E-1009, F-1040, G-1074) and five greases (H-1027, H-1040, H-1081, J-1012, J-1074), using tests of twenty drops from drop heights of 43.3, 30, and 15 inches.

TABLE 5. RELATIVE SENSITIVITIES AND REACTION
INTENSITIES OF VARIOUS GREASES

<u>Sample</u>	<u>50-Percent Point, in.</u>	<u>Avg Peak Press., psi</u>	<u>Max Peak Press., psi</u>
F-1018	8.0	11.8	33.6
H-1027	14.3	5.0	16.3
H-1040	12.8	0.47	3.62
H-1081	12.2	2.1	5.9
	9.4	1.0	1.8
	10.3	1.6	6.7
	10.3	1.4	6.2
	13.3	3.3	7.4
H-1085	18.2	0.16	0.29
	10.6	0.06	0.16
	14.5	0.14	0.24
J-1012	15.2	0.05	0.16
	10.0	0.01	0.14
	14.5	0.33	2.52
J-1022	5.6	1.8	5.5
J-1053	11.7	16.4	21.0
J-1074	14.9	0.10	0.20
	15.9	0.04	0.20
	11.0	0.04	0.16
	13.4	0.11	0.19
	12.9	0.05	0.16

Note: Each test consisted of twenty-five drops made by the up-and-down method, using Assembly III.

TABLE 6. RELATIVE SENSITIVITIES AND REACTION
INTENSITIES OF VARIOUS LIQUIDS

<u>Sample</u>	<u>50-Percent Point, in.</u>	<u>Avg Peak Press., psi</u>	<u>Max Peak Press., psi</u>
D-1071	21.9	0.03	0.17
D-1093	14.7	2.9	11.1
	13.3	4.1	11.5
E-1005	10.9	2.7	6.4
E-1009	18.9	0.07	0.17
	16.4	0.06	0.45
E-1010	17.4	0.05	0.14
F-1040	20.0	0.00	0.00
G-1074	14.3	10.4	22.9
J-1050	16.4	26.5	64.8
J-1052	16.1	0.02	0.14
J-1080	13.2	20.5	46.2

Note: Each test consisted of twenty-five drops made by the up-and-down method, using Assembly III.

TABLE 7. RELATIVE SENSITIVITIES AND REACTION
INTENSITIES OF MIXTURES OF TWO LIQUIDS

<u>Sample</u>	<u>50-Percent Point, in.</u>	<u>Avg Peak Press., psi</u>	<u>Max Peak Press., psi</u>
E-1010	17.4	0.05	0.14
95% E-1010 - 5% J-1080	16.4	0.00	0.00
90% E-1010 - 10% J-1080	15.2	0.04	0.21
70% E-1010 - 30% J-1080	17.0	0.40	4.0
30% E-1010 - 70% J-1080	17.0	7.30	39.0
J-1080	13.2	20.50	46.2

Note: Each test consisted of twenty-five drops made by the up-and-down method, using Assembly III.

TABLE 8. REACTION FREQUENCIES AND INTENSITIES OBTAINED AT
THREE DROP HEIGHTS IN 20-DROP TESTS

Sample	43.3 in.			30 in.			15 in.		
	No. of Reactions	Avg Peak Press., psi	Max Peak Press., psi	No. of Reactions	Avg Peak Press., psi	Max Peak Press., psi	No. of Reactions	Avg Peak Press., psi	Max Peak Press., psi
E-1009	20	2.6	18.0	18	1.6	9.2	3	0.0	0.0
	18	4.9	18.1						
	20	2.7	16.2						
F-1040	20	0.1	0.4	20	0.0	0.0	0	0.0	0.0
	18	0.1	0.2						
	20	0.1	0.2						
G-1074	20	15.9	61.4	20	9.4	19.0	13	3.8	17.5
	20	12.5	29.5						
	20	15.9	71.4						
H-1027	20	11.0	36.9	20	4.2	10.5	8	3.3	7.4
	20	10.0	37.1						
	20	12.4	26.4						
H-1040	20	0.9	3.7	19	1.2	3.7	9	0.4	3.6
	19	1.7	6.2						
	20	0.5	3.8						
H-1081	20	5.7	8.0	20	2.8	5.7	20	3.2	7.0
	20	3.7	7.7						
	20	4.3	11.7						
J-1012	20	0.1	1.0	20	0.7	3.4	18	0.0	0.0
	20	0.7	3.3						
	20	0.6	3.6						
J-1074	20	0.2	0.4	20	0.1	0.2	0	0.0	0.0
	20	0.1	0.2						
	20	0.1	0.2						

Note: Assembly III was used.

It will be noted that the average and maximum peak pressure readings at both 43.3 and 30 in. were higher than those previously obtained for the same materials using the up-and-down test (Tables 5 and 6) which, of course, involved considerably lower drop heights. Note also that the general reaction intensity levels associated with each material at 43.3 in. and at 30 in. followed the same trends previously noted in the up-and-down tests. These distinctions were, however, not so precise at 15 inches. This was probably due to the several very low reaction frequencies obtained which, in turn, were most likely the result of being within the probability distributions associated with the 50-percent points of these materials.

As previously stated, the primary purpose of this test series was to check the repeatability of peak pressure measurements under controlled conditions. As can be seen, the separation of general intensity levels noted in the previous up-and-down tests was confirmed by the tests made at 43.3-in. and 30-in. drop heights. Further, as might have been inferred from the 50-percent points of the various materials, both the 43.3-in. and 30-in. drop heights produced near 100-percent reaction frequencies. Under these conditions, the greatest amount of peak pressure data would be collected and averaged, and the best repeatability of the average and maximum peak pressures would be obtained. The degree of repeatability, as can be seen, was generally very good, particularly in relation to the general levels of average and maximum peak pressures to be differentiated. Accordingly, it was concluded, from the standpoints of the efficiency of the test and the repeatability of the results, that peak pressure measurements conducted at a fixed, rather high drop height (say, 30 to 43.3 in.), would be most desirable.

Having reached the conclusion above, it was then decided to rerun the earlier tests on mixtures of E-1010 and J-1080 liquids using, instead of the up-and-down procedure, a fixed drop height of 43.3 inches. The results are given in Table 9. Comparison of Tables 7 and 9 shows that the trends are basically similar.

c. Effect of Anvil Region Assembly and Sample Thickness

The two preceding test series were made with Anvil Region Assembly III and a sample thickness of 0.020 in. by virtue of the specimen cup design. In order to evaluate the effects of such variables as the design and materials of specimen cups and striker pins and the thickness of the test sample, additional tests were performed using the Bulletin 527 anvil region assembly, with sample thicknesses of 0.020 and 0.050 inch. These tests were made on the same eight liquids and greases used in the second test series, at the same drop height of 43.3 in., and likewise at twenty drops per test. Table 10 compares the results of these tests (Cases B and C) with those obtained in the second test series (Case A). Obviously, Case A was far superior to Cases B and C at depicting potential hazards. For example, H-1027 gave no reactions at all in Case C and rather infrequent and mild

TABLE 9. REACTION INTENSITIES OF MIXTURES OF
TWO LIQUIDS AT 43.3-IN. DROP HEIGHT

<u>Sample</u>	<u>Avg Peak Press., psi</u>	<u>Max Peak Press., psi</u>
E-1010	0.2	0.2
90% E-1010 - 10% J-1080	0.2	0.4
80% E-1010 - 20% J-1080	0.4	1.7
70% E-1010 - 30% J-1080	1.8	3.8
50% E-1010 - 50% J-1080	7.7	19.0
30% E-1010 - 70% J-1080	14.0	38.0
J-1080	9.5	44.7

Note: Each test consisted of twenty drops, using Assembly III.

TABLE 10. REACTION FREQUENCIES AND INTENSITIES OBTAINED
WITH THREE ANVIL REGION-SAMPLE THICKNESS COMBINATIONS
IN TWENTY DROPS FROM 43.3 INCHES

Sample	Case A*			Case B†			Case C†		
	No. of Reactions	Avg Peak Press., psi	Max Peak Press., psi	No. of Reactions	Avg Peak Press., psi	Max Peak Press., psi	No. of Reactions	Max Peak Press., psi	
E-1009	20	2.6	18.0	6	2.7	16.2	1	0.2	
F-1040	20	0.1	0.4	1	0.0	0.0	0	0.0	
				0	0.0	0.0			
				0	0.0	0.0			
G-1074	20	15.9	61.4	5	24.8	39.0	2	3.7	
H-1027	20	11.0	36.9	1	0.0	0.0	0	0.0	
				5	0.6	1.5			
H-1040	20	0.9	3.7	2	0.2	0.4	1	0.8	
H-1081	20	5.7	8.0	9	0.1	0.4	0	0.0	
J-1012	20	0.1	1.0	3	0.5	1.6	0	0.0	
J-1074	20	0.2	0.4	0	0.0	0.0	0	0.0	

*Using Assembly III.

†Using Bulletin 527 anvil region assembly and 0.020-in. sample thickness.

‡Using Bulletin 527 anvil region assembly and 0.050-in. sample thickness.

reactions in Case B but consistent and violent reactions in Case A. Even for an extremely violent reactor such as G-1074, substantially less frequent and less violent reactions were obtained in Cases B and C than in Case A.

d. Effect of Shock Wave Probe Location

All of the preceding work was performed with the shock wave probe so located that its inlet end was 2.375 in. from the nearest edge of the specimen cup. In an effort to obtain somewhat higher peak pressure readings, some tests were made with the probe moved closer to the reaction area so that the inlet end was 1.250 in. from the nearest edge of the specimen cup. It was thought that any expansion of the relative intensity scale might be helpful in better resolving the differences among various materials.

A series of 20-drop tests was made from a drop height of 43.3 inches. The results obtained are compared with the previous results in Table 11. It is seen that significant magnification of the peak pressure scale was achieved by moving the shock wave probe closer to the reaction area. Therefore, all subsequent tests were made using the 1.250-in. distance.

e. Effect of Small Changes in Transducer Location

There was some concern about the possible effects of small changes in the location of the transducer relative to the shock wave probe upon its replacement after cleaning operations, etc. If such effects should be significant, then, as can be seen from the geometrical relationship of the transducer and probe in Figure 7, rather sophisticated relocation procedures would have to be developed.

The procedure used in checking these effects involved taking sets of simultaneous measurements on a single shock wave at two points opposite each other and equidistant from the end of the probe. Then, more sets of simultaneous measurements were taken with one of the points moved away from its original location to simulate a relocation error. The distance moved would far exceed the most gross relocation error to be normally expected.

Figure 10 presents simultaneous shock wave traces obtained with two newly purchased Kistler 606L transducers placed opposite each other with centers 0.5 in. from the end of the shock wave probe. The diaphragm faces were parallel with and set back 0.10 in. from the inside wall of the probe. It can be seen that each reaction produced very nearly the same signal wave form and magnitude in both transducers, as was to be expected.

Figure 11 shows traces obtained as above except that the transducer producing the bottom signal on each trace was moved 0.31 in. further from the end of the shock tube in the direction of the tube axis. It will be noted that, again, both transducers produced close to the same wave forms

TABLE 11. REACTION INTENSITIES OBTAINED WITH INLET
END OF SHOCK WAVE PROBE LOCATED AT DIFFERENT
DISTANCES FROM REACTION AREA

Sample	1.250-in. Distance		2.375-in. Distance	
	Avg Peak Press., psi	Max Peak Press., psi	Avg Peak Press., psi	Max Peak Press., psi
J-1074	0.3	0.5	0.2	0.4
E-1010	0.2	0.4	0.2	0.2
90% E-1010 - 10% J-1080	0.2	0.4	0.2	0.4
70% E-1010 - 30% J-1080	1.2	7.5	1.8	3.3
J-1080	33.5	179.0	9.5	44.7

Note: Each test consisted of twenty drops from 43.3 in. using Assembly III.

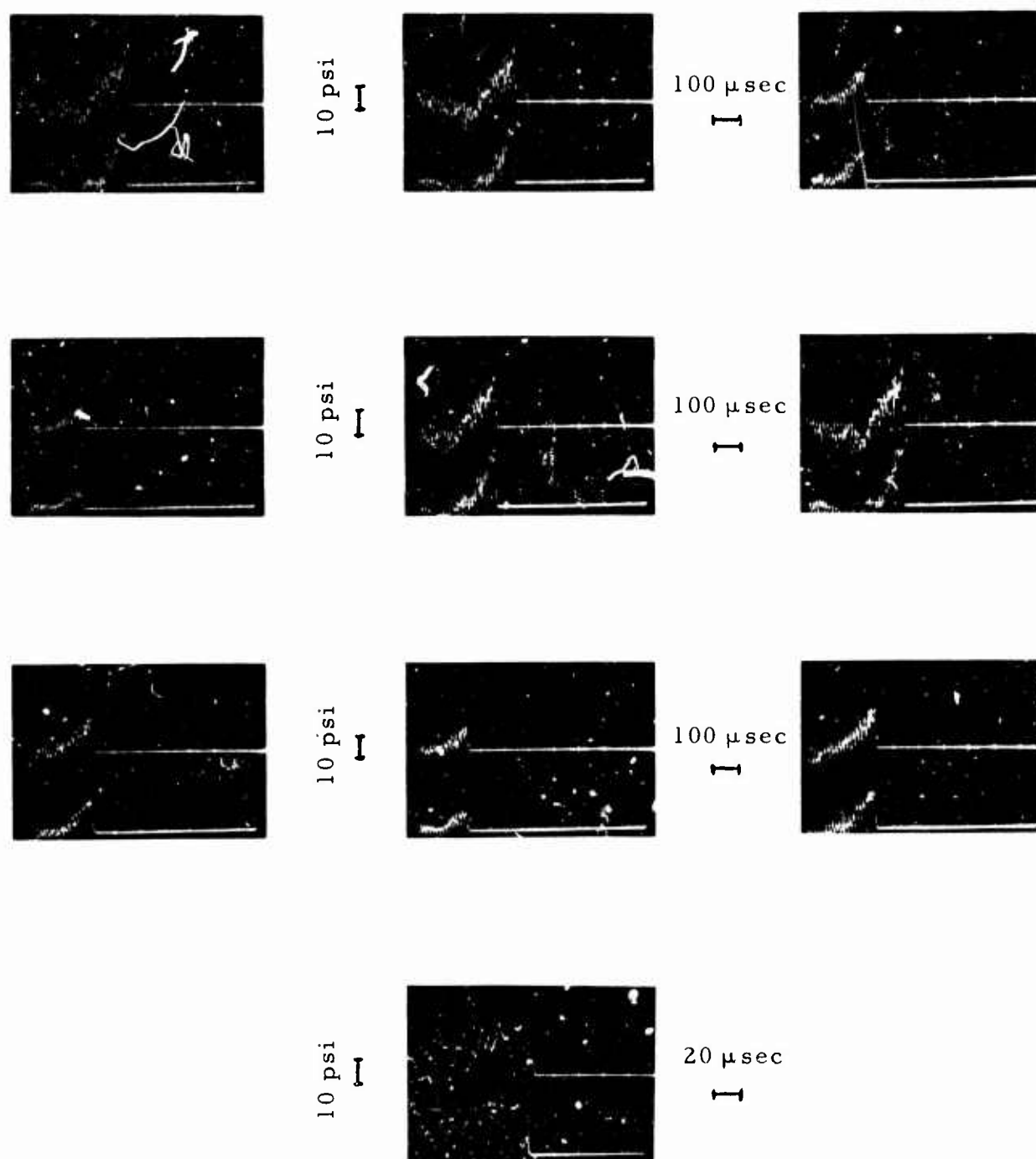


FIGURE 10. DUAL TRACES OBTAINED WITH BOTH TRANSDUCERS EQUIDISTANT FROM SHOCK WAVE PROBE

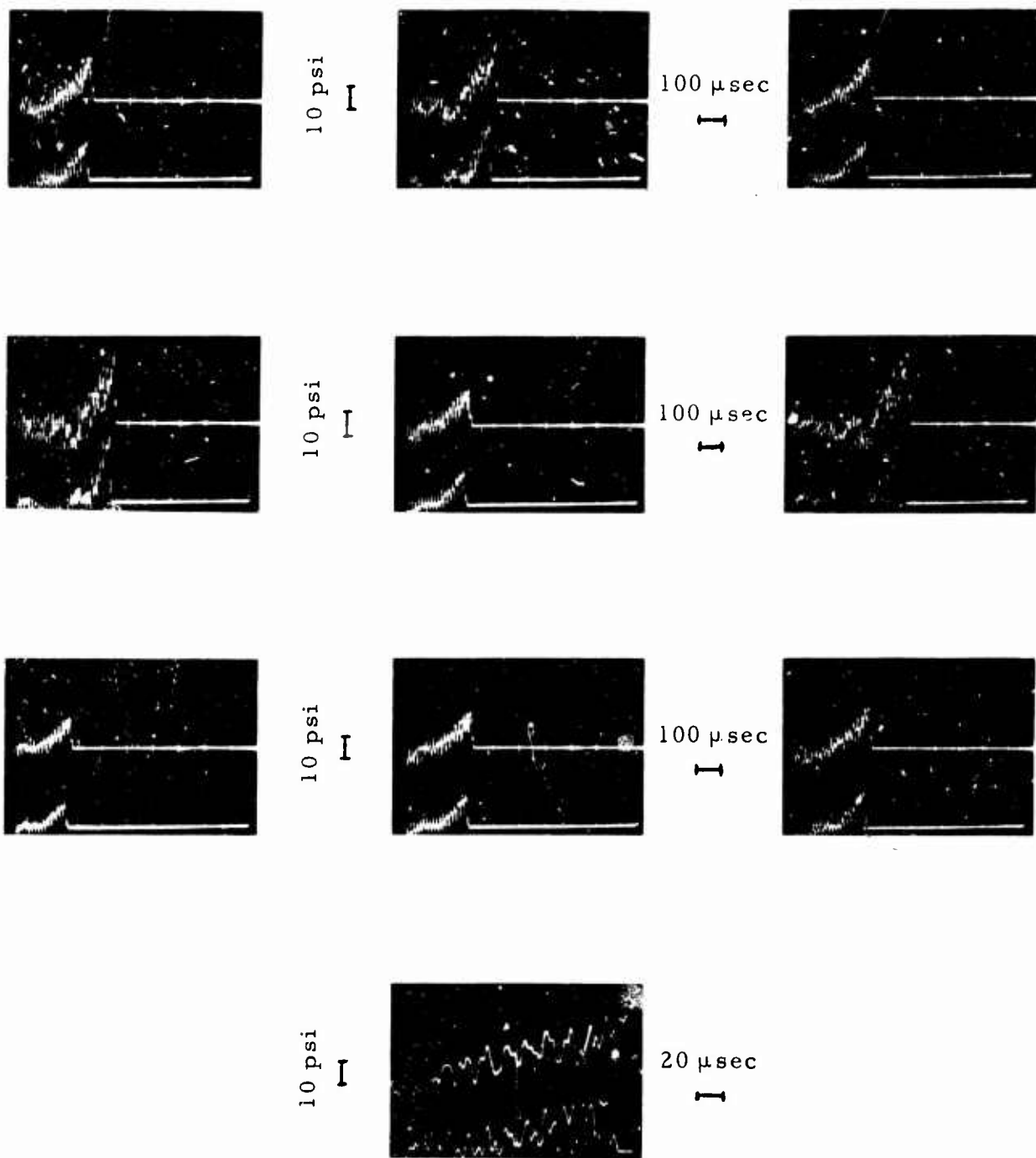


FIGURE 11. DUAL TRACES OBTAINED WITH THE LOWER
TRANSDUCER 0.31 IN. FURTHER FROM
SHOCK WAVE PROBE

and peak magnitudes for any one reaction. It was concluded from these results that location of the transducer was not extremely critical with respect to the distance from the end of the tube. In other words, extremely close tolerances would not be necessary in replacing transducers after cleaning, etc.

A question has probably arisen in the mind of the reader at this point with respect to why these traces show so much ringing noise in comparison with the traces shown earlier. This question will be taken up later (Section III.3) since its discussion here would involve a rather lengthy digression.

f. Investigation of Low-Intensity Reactions

There was some question as to whether the low-intensity registrations indicated by the peak pressure measuring system were indeed reactions or some other phenomena. In an effort to resolve this question, tests were conducted on two greases--an inert grease J-1074 and a moderately reactive grease H-1081--with liquid nitrogen and liquid oxygen, respectively, as the cryogenic fluid. Each test consisted of twenty drops from a drop height of 43.3 inches. The shock wave probe was located 1.25 in. from the nearest edge of the specimen cup, as in the fourth test series.

Table 12 shows that, although H-1081 showed considerably different behaviors in liquid nitrogen and liquid oxygen, the results on J-1074 were substantially the same. One might be tempted to conclude that, with nitrogen being inert, the low-intensity registrations could have been due to something other than reactions. However, it must be remembered that it is very hard to prepare grease samples without entrapping tiny bubbles of air which of course contain oxygen. Further, liquid nitrogen exposed to air is constantly condensing oxygen from the air so that the surface of the nitrogen will be composed, to some degree, of liquid oxygen. This layer of liquid oxygen can be pushed down to the surface of the sample when the striker pin is lowered. Considering these factors, it becomes apparent that the tests were not conclusive since oxygen was in fact not necessarily excluded.

It was decided to investigate whether such low-intensity indications actually came from pressure pulses passing up the shock probe tube or from vibrations from the impacting plummet reaching the transducer through its mount. To do this was simply a matter of placing a cap over the transducer diaphragm. In ten drops on J-1074 with the transducer capped, no registrations were obtained. In ten subsequent drops on J-1074 with the cap removed, results quite similar to those on J-1074 in Table 12 were obtained. It was, therefore, concluded that a low-intensity registration was a valid indication of the passage of a shock front through the tube rather than an indication of some spurious signal.

The question of whether the shock front passing through the tube is imparted by a chemical reaction in these "low-intensity" cases remains

TABLE 12. REACTION FREQUENCIES AND INTENSITIES
OBTAINED IN LIQUID NITROGEN AND LIQUID OXYGEN

<u>Sample</u>	<u>Cryogenic Fluid</u>	<u>No. of Reactions No. of Drops</u>	<u>Avg Peak Press., psi</u>	<u>Max Peak Press., psi</u>
H-1081	Nitrogen	20/20	0.1	0.3
H-1081	Oxygen	20/20	20.6	36.8
J-1074	Nitrogen	20/20	0.1	0.2
J-1074	Oxygen	20/20	0.1	0.3

Note: Each test consisted of twenty drops from 43.3-in. drop height.
Assembly III was used.

to be answered since no clearcut method has been found for determining whether a minute reaction has or has not occurred. Many cases have been observed in which low-intensity readings (in the range of about 0.10 to 0.80 psi) have manifested definite evidence of reaction, such as distinct char. On the other hand, many other low-intensity readings in the same range have been accompanied by no discoloration at all, with the only evidence being erosive patterns in the sample remains, as if a gas had expanded rapidly. Still further, some materials have produced loud reactions while leaving no discolored residue, showing that char is not a final arbiter.

It should be emphasized that, in measuring impact sensitivity, the sometimes rather ambiguous reaction criteria form the only basis for the operator decisions, which in turn cannot but influence the rating results. Conversely, operator judgment is of little importance in reaction intensity measurement; whether a reaction has or has not occurred is little more than academic as long as the reaction intensity is of a very low order.

6. EXPLORATORY REACTION INTENSITY MEASUREMENTS ON SOLIDS

The up-and-down test results on acetate, phenolic, and Mylar reported earlier in this report showed that the 50-percent drop heights of solid materials were quite widely separated. It was also shown in the last summary report⁽¹¹⁾ that the logarithm of the 50-percent point of acetate varied linearly with the striker pin surface area and that the configuration of the striker pin and specimen cup had little influence on sensitivity, at least for the material tested. This behavior of a solid is apparently quite different from the behaviors of greases and liquids just discussed. In an effort to gain some idea as to how the reaction intensity of solid materials would behave, tests of twenty drops each were performed on acetate and phenolic of 0.020-in. thickness.

Table 13 presents the results obtained. It will be noted that both samples produced considerably lower reaction frequencies at 43.3 in. when using larger diameter striker pins than when using smaller diameter striker pins. This is not surprising because of the relationship between 50-percent point and striker pin surface area noted previously.

It was reasoned earlier that, in the interests of the efficiency of the test and the repeatability of the test results, it would be desirable to conduct reaction intensity measurements at nearly 100-percent reaction frequency. Granting this, it would then appear that reaction intensity tests on solids might need to be conducted with a smaller striker pin diameter (such as 0.7 in.) than that used in tests on liquids and greases (0.855 in.). Further, considering the apparent significance of striker pin surface area in the case of solids, it would be desirable to evaluate the sensitivity of solids using different striker pin diameters. This is somewhat of a complication, for it is generally preferable if all materials can be evaluated with the same test apparatus, specimen cups, and striker pins. However, from the standpoint of test

TABLE 13. REACTION FREQUENCIES AND
INTENSITIES OF SOLIDS

<u>Sample</u>	<u>Striker Pin Diameter, in.</u>	<u>Drop Height, in.</u>	<u>No. of Reactions No. of Drops</u>	<u>Avg Peak Press., psi</u>	<u>Max Peak Press., psi</u>
Acetate	0.5	43.3	18/20	2.1	15.7
	0.7	30.0	18/20	3.8	17.8
	0.7	43.3	20/20	5.9	10.4
	0.855	43.3	7/20	0.9	4.6
	0.855	48.0	9/20	3.1	5.7
Phenolic	0.5	43.3	20/20	7.2	16.1
	0.7	43.3	19/20	23.1	41.1
	0.855	43.3	12/20	3.5	19.6

Note: Each test consisted of twenty drops from specified drop height.
Assembly III was used.

efficiency and repeatability, the use of a single diameter striker pin for liquids and greases and different diameter striker pins for solids appears to be warranted, considering that only the striker pin guide need be changed when a different diameter striker pin is used.

7. DISCUSSION OF TEST METHODS

The intent of this discussion is to review in summary fashion the rather voluminous data already generated at this juncture and to consider the outlook of the various test methods. For the sake of convenience, it is proposed first to compare the results of various test methods on liquids and greases on which the greatest amount of data has been accumulated. Following this, brief remarks will be made relative to the evaluation of solids. Finally, a discussion will be made of the tentative conclusions reached at this point in the research program which led to the test apparatus and procedure standardization and subsequent data generation reported in Section IV.

a. Test Methods for Liquids and Greases

Figure 12 presents threshold values obtained on three liquids (E-1009, F-1040, G-1074) and six greases (H-1027, H-1040, H-1081, J-1012, J-1053, J-1074), using Bulletin 527 equipment. In this and the two subsequent figures, the letter suffix "P" below the sample code indicates that the sample has passed the Bulletin 527 test, and the letter suffix "F" denotes that the sample has failed the Bulletin 527 test. It is seen that the repeatability of the threshold values was poor, particularly for the highly reactive H-1027 which, incidentally, was passed by the Bulletin 527 method at 42 inches. This instance alone indicates that the validity of the threshold value determination is questionable.

Further evidence of the questionable nature of this type of test method is evident in the following precision statement released by ASTM⁽⁹⁾:

<u>Threshold Value Drop Height, in.</u>	<u>Repeatability, One Operator and Apparatus</u>	<u>Reproducibility, Diff. Operators and Apparatus</u>
24	17.0	40.8
15	10.6	25.2
6	4.3	10.1

This precision statement was generated from data gathered in two independent round-robin test programs which included a number of laboratories having a capability in this test. It demonstrates the level of precision of which the threshold value test method is currently capable.

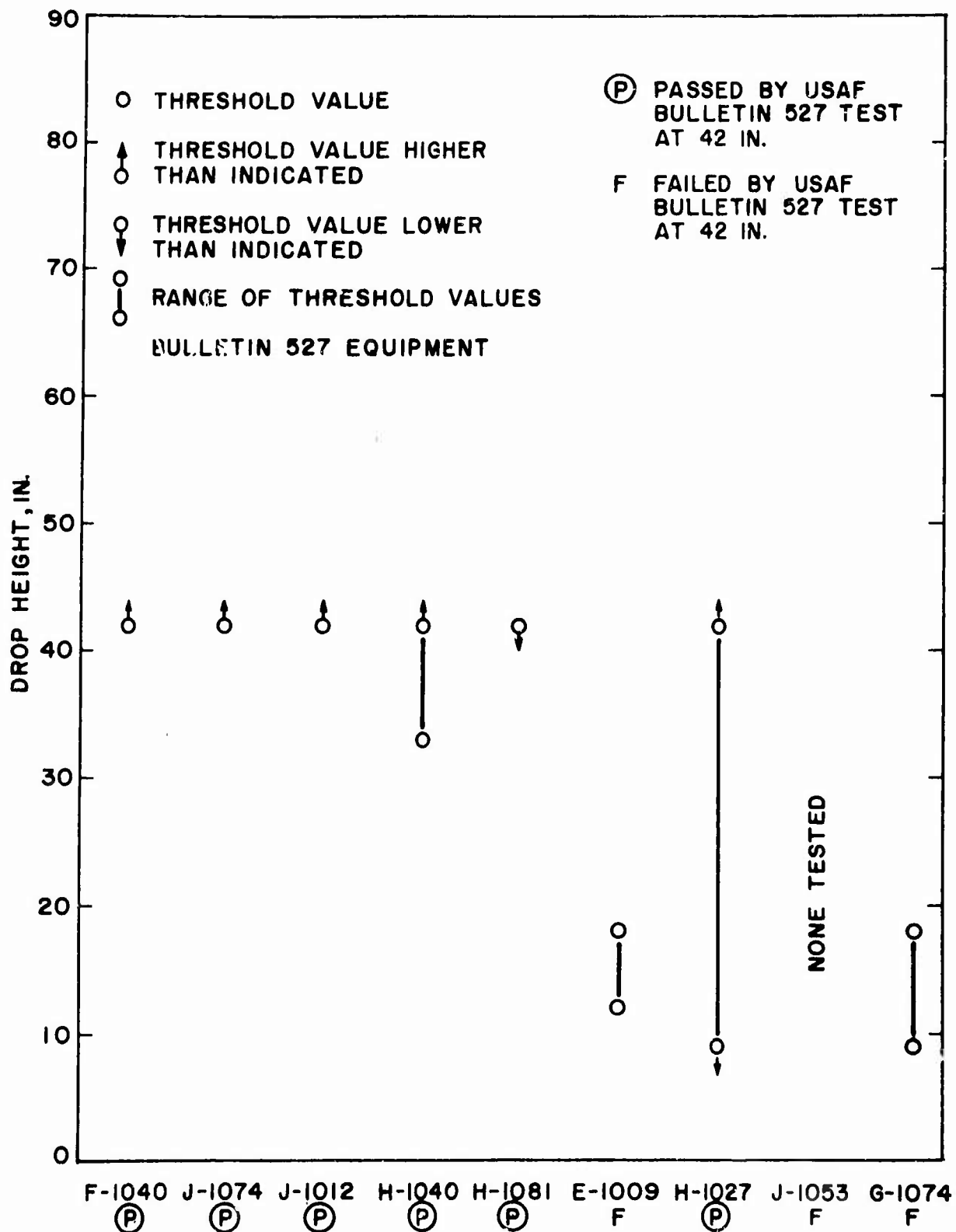


FIGURE 12. TYPICAL THRESHOLD VALUES OF LIQUIDS AND GREASES

It can be seen that the ASTM data cast considerable suspicion upon the present practice of passing a material if it survives twenty drops at 42 or 43.3 in. without reaction, as it appears that such a single "threshold value" could easily be obtained on a material having an intrinsic threshold value considerably lower. Furthermore, one wonders about cases in which certain batches of a material passed such tests while other batches failed, with this anomaly being explained as batch-to-batch variance of sensitivity. Did the sensitivity of the material vary or was it the variance obviously inherent in the test method?

The 50-percent point determinations on the nine materials of Figure 12 are presented in Figure 13. These tests were made using Assembly III and the up-and-down test which, as discussed in Section I, is statistically far more reliable than threshold value type methods. Here, it is seen that the 50-percent points of the various materials are very nearly the same within the limits of repeatability of this test. In particular, the 50-percent points of all six greases are in one band about 6 in. wide, and the three liquids are in another band of about the same width. It appears, therefore, that liquids and greases cannot be confidently separated for possible service on the basis of their sensitivities. Further, it appears that many of the sensitivity differences previously indicated by threshold value methods were indeed characteristics of the test methods rather than of the materials.

Finally, Figure 14 presents the results of reaction intensity measurements on the various materials, using Assembly III and a drop height of 43.3 inches. In assessing the ability of this test method to separate materials, it is first necessary to define some separation criteria that might logically apply to service conditions as well as to this bench test. Such a definition requires simultaneous consideration of several factors as will be discussed in the following paragraphs.

One of these factors is concerned with the ignition stimulus as it occurs in a drop test. In work described in this and the previous report⁽¹¹⁾, it was shown that ignition was more dependent upon the striker pin-specimen cup configuration and the physical state of the sample than upon the impact energy delivered. Additional proof of this has been gathered by conducting up-and-down tests with different plummet weights. Using a plummet weighing 7.75 lb and Assembly III in a 50-drop test, a 50-percent point of 13.1 in. was obtained for G-1074. This may be compared with the average 50-percent point (ten 25-drop tests, Table 3) of 14.6 in. obtained using the standard 20-lb plummet. It is apparent that, in this case, the 61-percent reduction in plummet weight had very little effect on the sample's sensitivity. Considering this, it is not unreasonable to assume that ignition could be achieved under a number of different combinations of impact energy and cup-pin configuration or, in service, under many different combinations of impact energy and

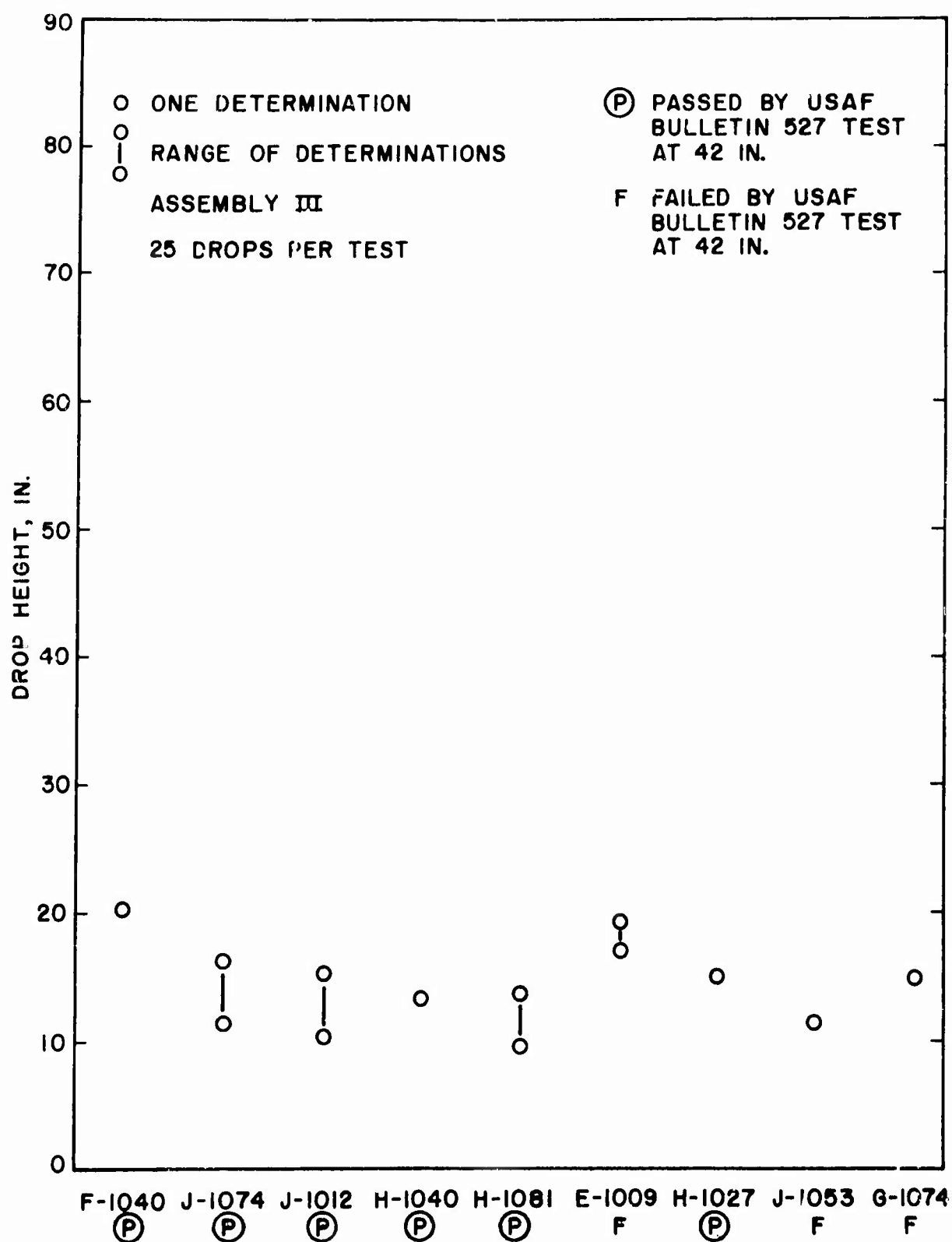


FIGURE 13. TYPICAL 50-PERCENT POINTS OF LIQUIDS AND GREASES

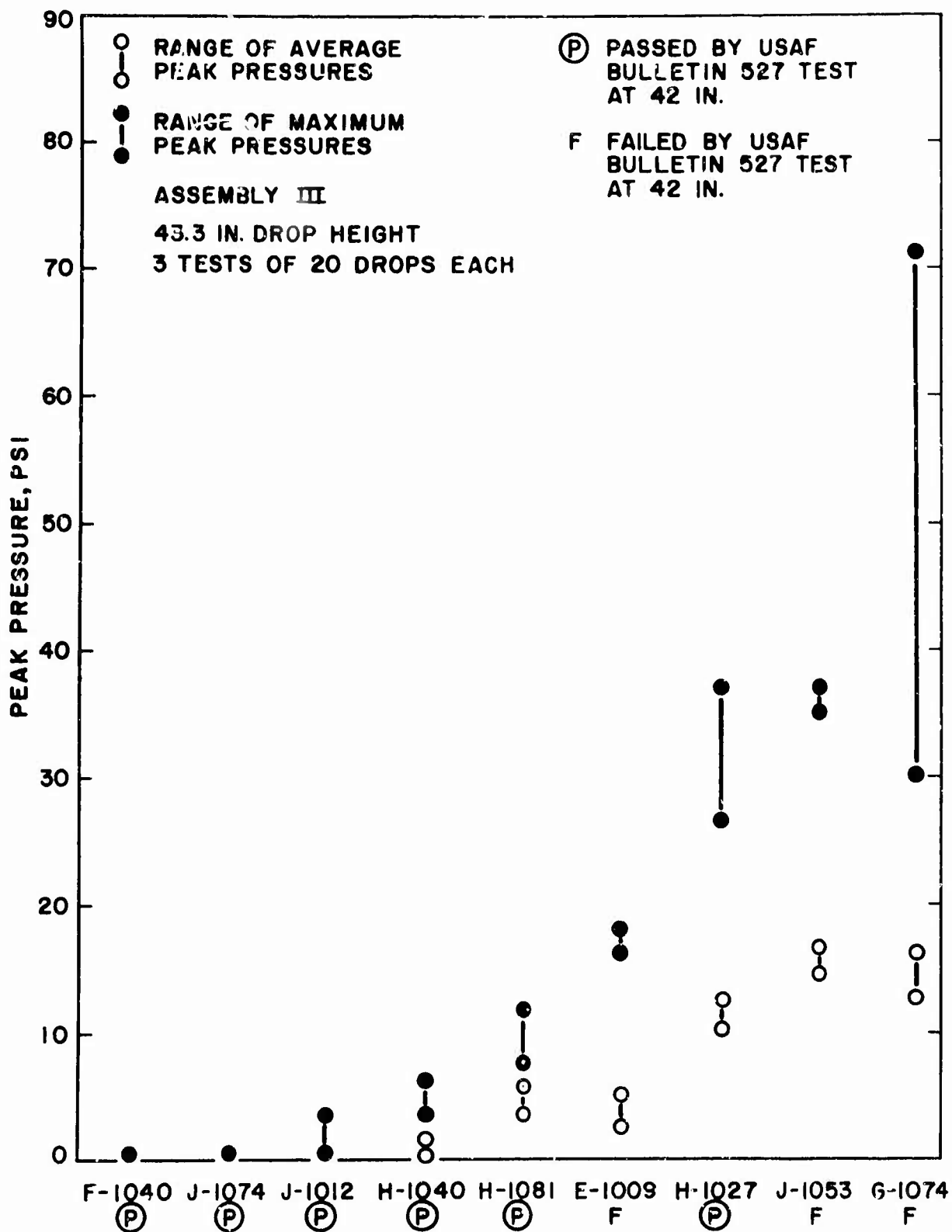


FIGURE 14. TYPICAL REACTION INTENSITIES OF LIQUIDS AND GREASES

Another factor to be considered is that liquids and greases are subjected to a wide variety of service conditions. For example, a frozen piece of thread sealant may be carried by liquid oxygen into a valve or into a pump where it may be trapped between moving parts or may be impacted by hydraulic surge. In any case, considering the many different possibilities of material size, material entrapment, impact energy, etc., it appears that it would be very difficult, if not impossible, to predict whether a combination of these parameters sufficient for ignition could occur.

Still another question is whether mere initiation of a reaction is indeed the criterion of primary interest. In the literature on this test, the words compatibility, reactivity, and sensitivity seem to be used as though they were interchangeable, and the connotation seems to be that if a reaction occurs in service, disaster must be the consequence. There is considerable doubt that reaction as such is necessarily hazardous, particularly in view of the rather ambiguous definition of a reaction. There is no doubt that self-propagating reactions are potentially dangerous, but there is considerable doubt whether nonpropagating reactions (such as those evidenced by slight char but not accompanied by visible flash, audible sound, or distinct shock waves) are indeed dangerous. Bearing in mind the difficulties in identifying service conditions, it is conceivable that some nonpropagating reactions might have occurred without resulting in damage and thus might have passed unnoticed. On the other hand, if explosions have occurred in service, one should find it hard to accept the premise that the reactions were, by their very nature, not self-propagating.

Judging from the above factors, it appears that a reliable test method would assume that a material could possibly react in service and that the rating would be on the basis of whether the reaction could lead to system damage, i. e., on the basis of the intensity of reaction propagation. This means that the separation criterion for the reaction intensity test will have to be that peak pressure reading corresponding to a reaction intensity sufficient to cause system damage. Now, obviously, judgment and experience will have to be brought to bear in the selection of this point. For the purpose of analyzing the results in Figure 14, however, it can be said that reactions become definitely apparent (flash, audible report, etc.) at peak pressures somewhere between 1.0 and 2.0 psi, while those above 2.0 psi are so violent that they must be regarded as explosive.

In Figure 14, as in the previous tables, both average and maximum peak pressure readings are shown. However, we are interested primarily in the potential hazard associated with a material, and the maximum peak pressure reading is the best indicator of this. On this basis, it can be seen that seven of the nine materials yielded at least one explosive reaction and therefore must be rated as potentially hazardous. Also, it will be noted that this test was successful in identifying the potential hazard of four different materials (J-1012, H-1040, H-1081, and H-1021) which had previously passed the Bulletin 527 test at 42 in. without reaction.

In evaluating the performance of the reaction intensity test, there are two other factors which merit consideration. First, the reaction intensity test depends on a quantitative measure of the peak shock wave pressure; as such, it leaves human factors completely out of the realm of the data-gathering process. Second, and just as important, is the fact that, even though impact-induced reactions are not repeatable, the very nature of the reaction intensity test can accommodate the statistics if the data are suitably interpreted. For example, the average peak pressure is not a suitable criterion because it does not respond to the most potentially hazardous situations; in effect, it averages out the highest value of the peak pressure readings from, say, twenty replicate drops--and these individual peak pressure readings, as shown earlier, do vary from drop to drop. On the other hand, by using the highest of all of the peak pressure readings as the criterion, the most potentially dangerous condition is identified with a high level of confidence.

b. Test Methods for Solids

In the case of solids, the limited data available at this point suggest that the degree of hazard could not be fully defined without measuring both impact sensitivity and reaction intensity. As in the case of liquids and greases, this tentative conclusion was reached by considering both test data and service conditions.

It has been shown for at least one solid material that, in contrast with liquids and greases, sensitivity varied significantly with the surface area of the striker pin under a given impact weight and that the pin-cup configuration had little influence. With this in mind, note that the service conditions seen by solids are also quite different from those seen by liquids and greases with their service being generally confined to usage as seals, gaskets, diaphragms, and valve seats. This suggests that, in many cases, these materials will stay in one piece and in one place during service. If this is so, then it may be possible to predict the shock parameters a given amount of solid material will experience and, ultimately, to correlate impact test results with service conditions for various solids.

On the other hand, there is always the danger that even a valve seat might be subjected to an unexpectedly severe stimulus. For this reason, a solid material should also be tested for its reaction intensity to determine whether a potentially hazardous reaction propagation can result if reaction does occur.

c. Conclusions Concerning Subsequent Work

By this time, it was felt that the ultimately desired format for the impact sensitivity and reaction intensity tests was fairly well defined. Of course, further work was indicated in the area of equipment refinement, but since the changes planned would not affect the plummet or pin-cup

configuration, it was felt that the basic model of stimulus would not be changed. Therefore, it was decided to discontinue additional data gathering except that necessary to equipment and procedure evaluation. Then, with the equipment design "frozen" and the test procedures standardized, an intensive data gathering program would be initiated. With these data, the degree of repeatability of the new test techniques could be defined, and additional information could be obtained on the behavior of the various materials.

SECTION III

STANDARDIZATION OF TEST APPARATUS AND PROCEDURES

1. GENERAL

This section will be devoted to a discussion of a number of changes made on test equipment in the light of the work described in the previous section. Each of these changes was effected with the idea in mind of developing an efficient and easily reproduced setup. Further, some aspects of the test procedures have been refined on the basis of the experience gained.

2. MECHANICAL COMPONENTS

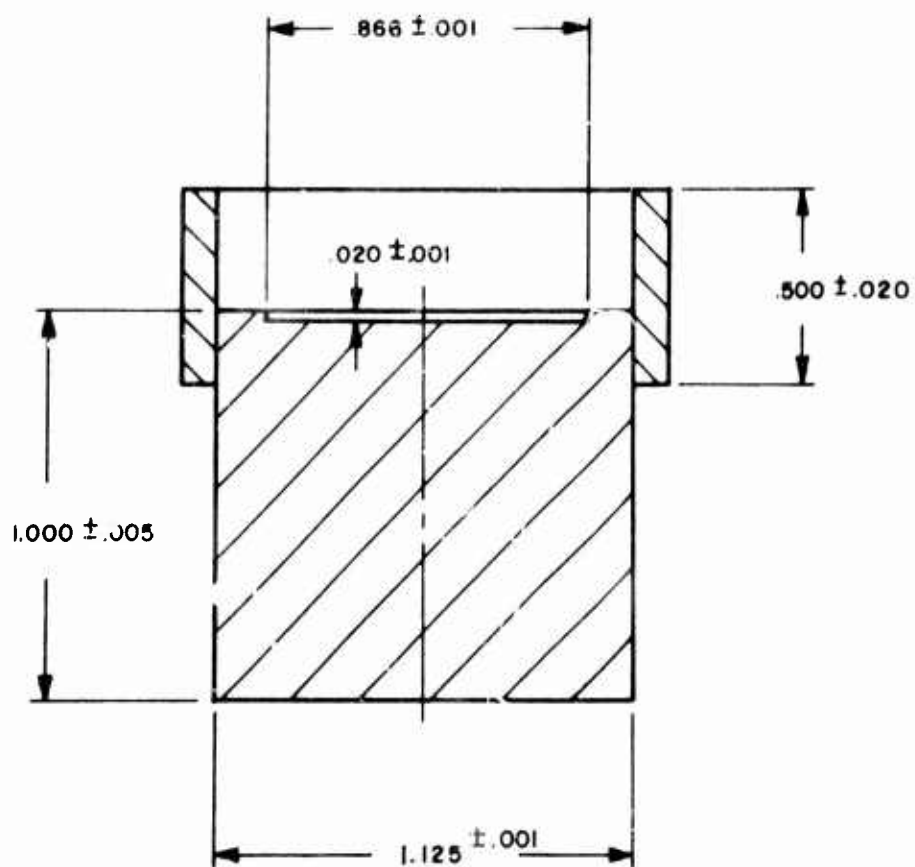
a. Specimen Cup and Sleeve

It was noted in the preceding tests that, because of the small clearance between the striker pin and the ID of the sample enclosure in the specimen cup, the striker pin would occasionally strike the side of the cup during impact. Further, in violent reactions, the upper edge of the specimen cup would occasionally break off from the forces of explosion. Finally, the upper Teflon sleeve would frequently break into pieces whenever the reaction was at all violent, and the flying debris would hit the surrounding walls.

To overcome these difficulties, a new specimen cup with an aluminum sleeve, shown in Figure 15, was introduced. Comparison of Figures 2 and 15 shows that the OD of the specimen cup was increased from 1.000 to 1.125 in., and the ID of the sample enclosure was increased from 0.860 to 0.866 inch. The aluminum sleeve was cut from stock 6061-T6 tubing of 1.250-in. OD and 0.065-in. wall thickness. A slight taper was machined on the upper OD of the specimen cup so that the sleeve could be lightly pressed onto the cup. This light press fit plus the increased coefficient of thermal expansion of aluminum over steel was found to provide an excellent seal at liquid oxygen temperature, much better in fact than that achieved by the Teflon sleeves. Cost of the aluminum sleeves proved to be considerably less than the Teflon sleeves with no apparent effect on test performance. Further, although the aluminum sleeve would often "barrel" and occasionally break during violent explosions, nevertheless, it would remain in the reaction area, and small pieces would only rarely break off to cause hazards.

b. Location of Shock Wave Probe

Based upon the findings discussed earlier, the shock wave probe was moved so that its inlet end was 1.250 in. from the nearest edge of the specimen cup. The general configuration remained the same as that shown in Figure 7.



SPECIMEN CUP:
MATERIAL:
 17-4 PH STAINLESS STEEL
HEAT TREAT:
 CONDITION H-900

SLEEVE:
MATERIAL:
 CUT FROM 6061-T6 ALUMINUM
 TUBING 1.250 O.D. X 0.065 WALL

FIGURE 15. STAINLESS STEEL SPECIMEN CUP
 WITH ALUMINUM SLEEVE

c. Introduction of Additional Shock Wave Probe

During the previously described investigation, it was noted that an occasional reaction would appear to the ear to be louder than the reaction intensity read would indicate. Also, upon post-impact examination, some samples would show clear evidence that the major part of the rapid gaseous expansion had occurred in a direction away from the shock wave probe. It took only a few tests with a second shock wave probe placed diametrically opposite the first probe to confirm that "directionality" of reactions was a factor to be considered.

Since any variance in peak pressure measurements due to directionality would be additive to that inherent in the propagation of the sample reaction, it was decided to try to reduce the influence of this variable insofar as possible. Ideally, of course, it would be preferable to utilize a number of shock wave probes with their inlets radially disposed at points equidistant from the reaction center. However, the guide rails and the rebound catcher had already preempted a good deal of space around the anvil region assembly, and additional space had to be reserved for the insertion and removal of specimen cups. Considering these limitations, it was decided to employ only one additional probe and measuring system. As can be seen in Figure 16, this second probe, a copy of the original, is placed diametrically opposite the original and at the same distance from the specimen cup.

3. ELECTRONIC COMPONENTS

a. Peak Pressure Meters

The peak pressure meter employed in all preceding work was designed to operate on batteries, and this caused some inconvenience in day-to-day operation. In addition, the response curve of the instrument departed somewhat from linearity in the low output region. To alleviate these inconveniences, a new peak pressure meter operating on 115-volt AC line voltage was fabricated and put into operation. As shown in Figure 17, this meter has a very near linear response. Figure 18 shows the circuit diagram for this meter. The introduction of the dual measurement system described above required, of course, two peak pressure meters of identical behavior.

b. Charge Amplifiers

Two new Kistler Model 504 charge amplifiers were employed to replace the Model 566 units in service heretofore. The "dial gain" feature of these new units allows the charge sensitivity of the transducer to be inserted in such a manner that the input to the transducer in psi will cause a specified output in volts from the amplifier. In addition, the new amplifiers make static calibration of the transducers quite simple.

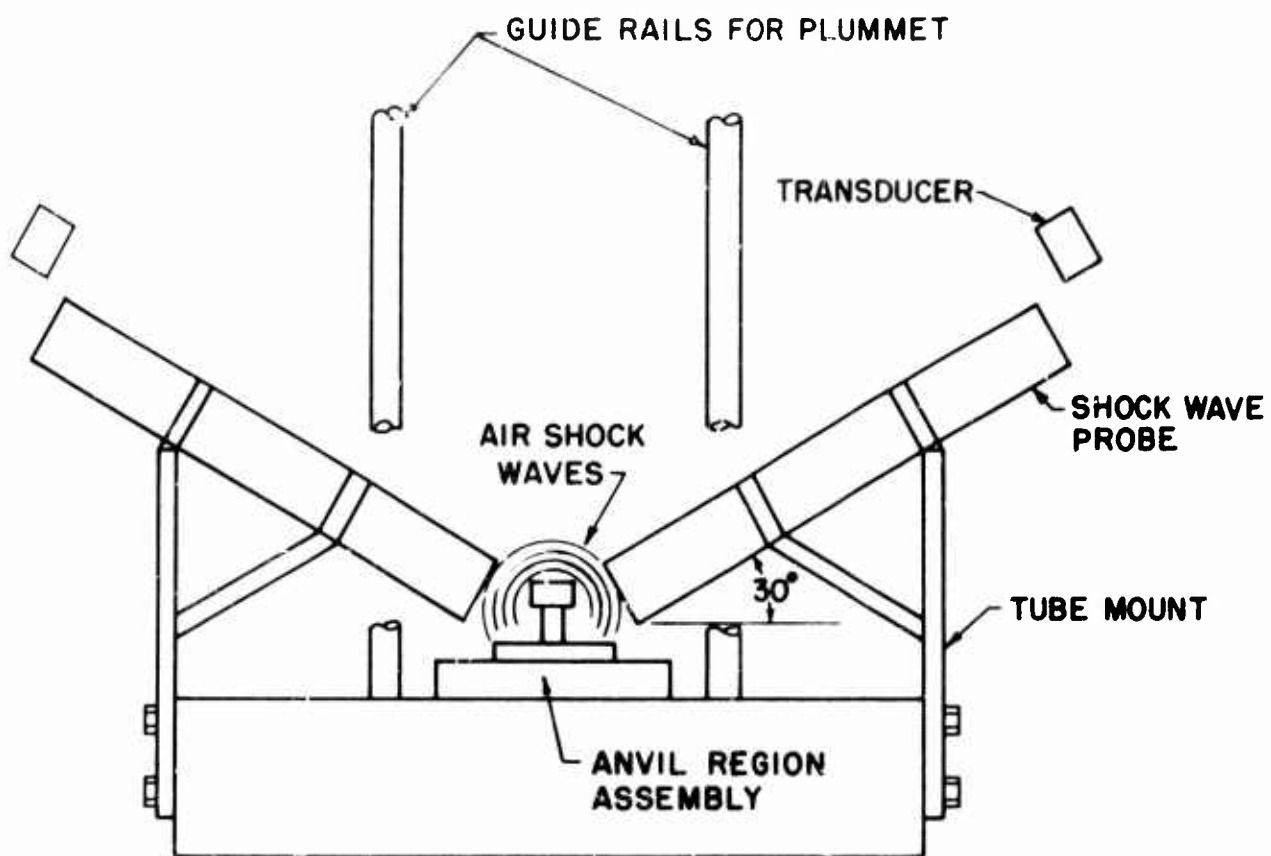


FIGURE 16. DUAL SHOCK WAVE PROBES

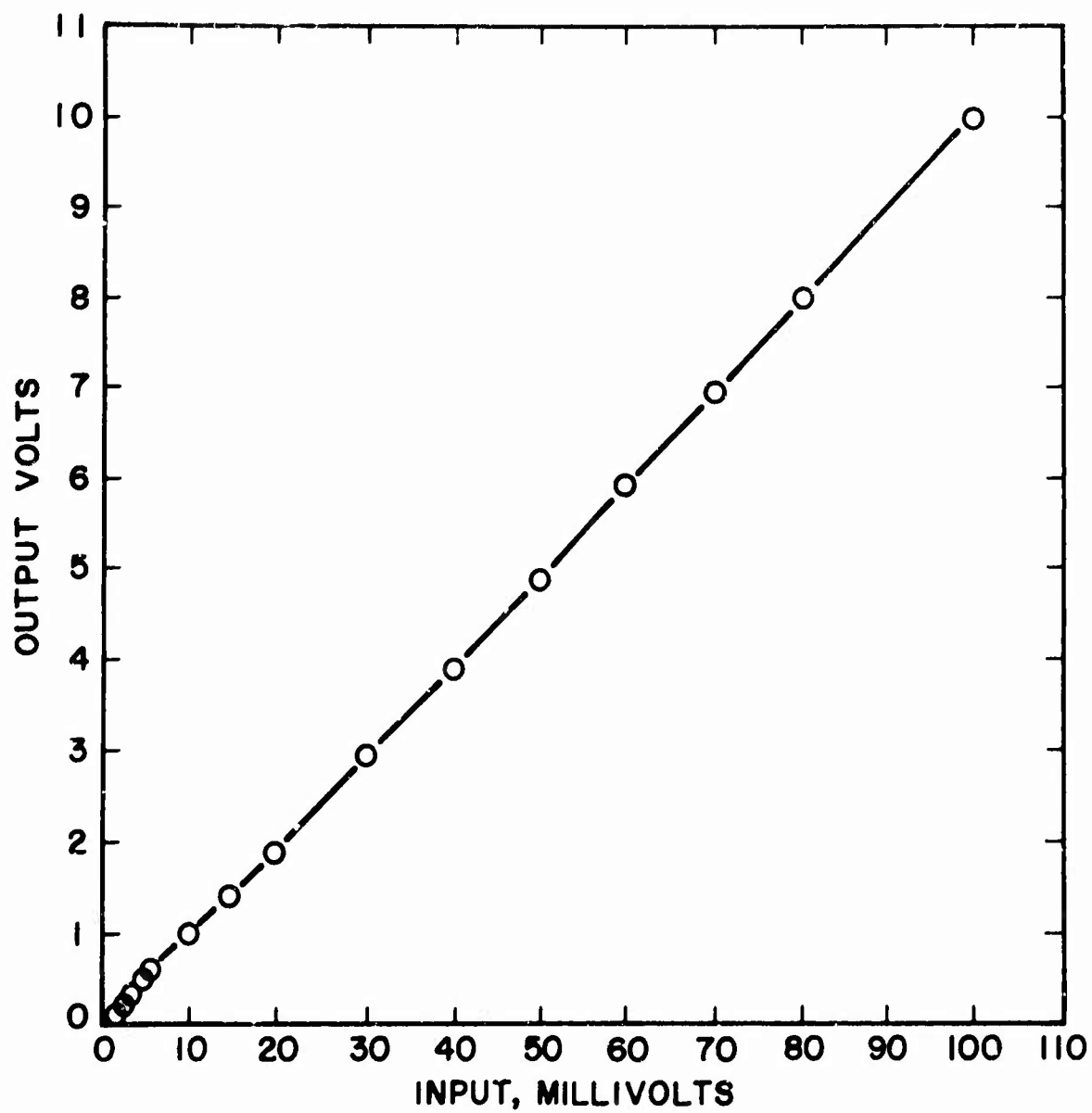


FIGURE 17. CALIBRATION OF PEAK PRESSURE METER

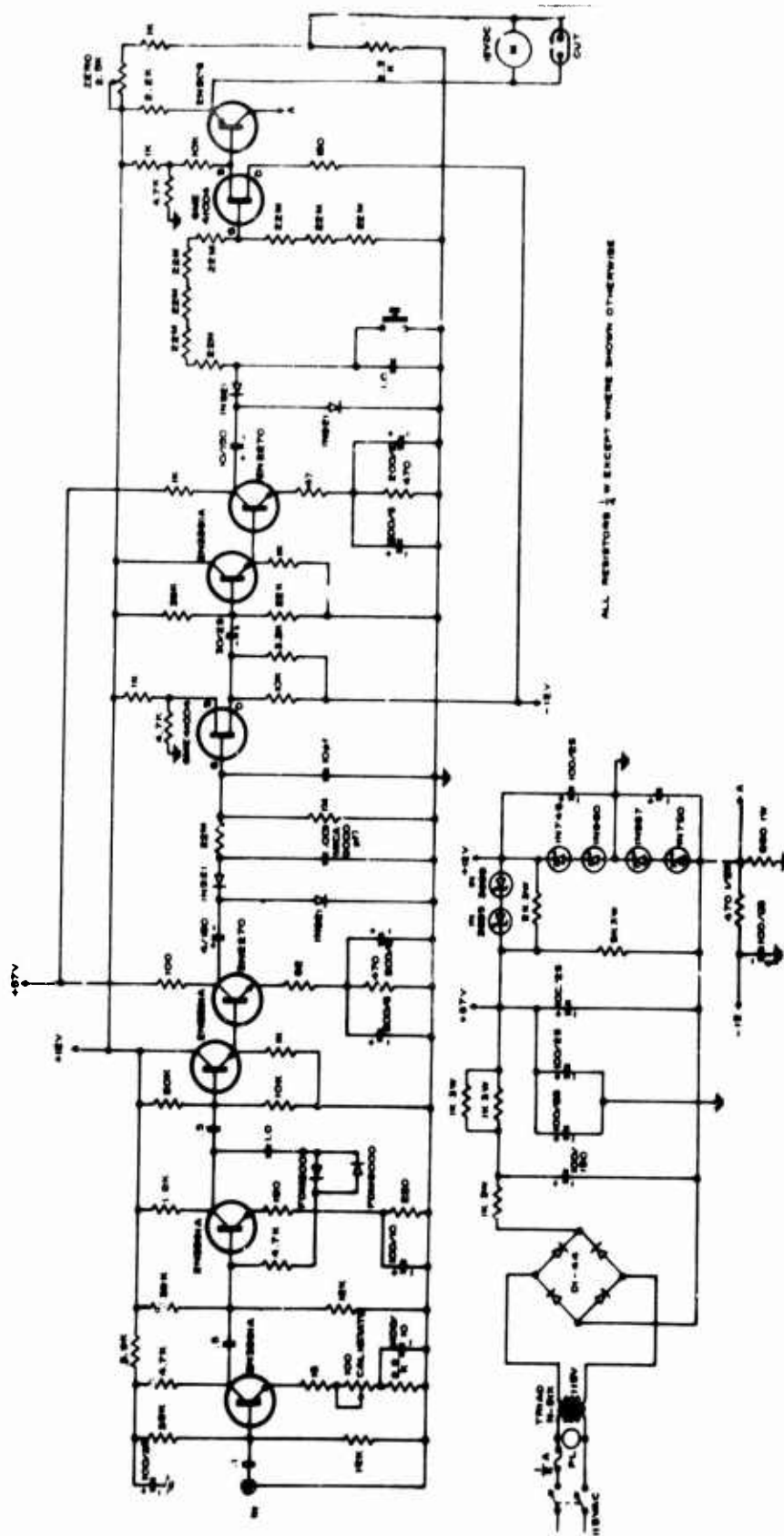


FIGURE 18. CIRCUIT DIAGRAM FOR PEAK PRESSURE METER.

c. Pressure Transducers

The previously used Kistler Model 601 pressure transducers were replaced by Kistler Model 606L transducers. The new transducers' output of approximately 5 picocoulombs (pcb) per psi, compared with the 0.3 pcb per psi output of the Model 601, allowed much better resolution in the pressure ranges of interest, particularly in the range of 0.1 to 1.0 psi.

It will be noted in the discussion attending Figures 10 and 11 that the 606L transducer showed greatly increased "ringing" compared with the 601, as in Figures 8 and 9. This generated some concern since the peak pressure meter would respond to the highest "ringing" peak which is considerably higher than the actual peak pressure in Figures 10 and 11 (the actual peak pressure reading would be at about halfway between the highest and lowest points of the first few ringing cycles). Other puzzling factors included the little "knee" which can be seen partway up the initial signal rise and the approximately 65-kc major ringing component shown even though the resonant frequency of the transducer was claimed by the manufacturer to be 130 kc.

After considerable experimentation, reasonable answers were found. First, with the transducer diaphragm parallel to the shock wave path (as it is when used with the shock wave probe), one shock wave was found to take about 15 μ sec to travel across the 606L's 0.25-in. wide diaphragm. Thus, it was reasoned that the transducer (rise time = 3 μ sec) must be responding before the shock wave could traverse the diaphragm, producing a waving effect that the acceleration compensation system in the transducer could not handle. It was also found that placing the diaphragm normal to the shock wave path caused the rise time to become much shorter, the "knee" to disappear, and the ringing frequency to increase to about 130 kc, which substantiated the above reasoning. Further, the comparatively "clean" traces shown by the 601 transducer were found to be the result of heretofore unsuspected poor high-frequency response of the Model 566 charge amplifier which acted as a high-frequency noise filter.

Paradoxically, noise filtration, undesirable in the previous case because of the source, turned out to be the solution to the ringing problem. By putting a 50-kc cutoff filter on the output of the 504 charge amplifier, the part of the ringing signal higher than the actual peak pressure can be greatly reduced. This can be seen in Figure 19 which shows a trace from a filtered 606L transducer signal.

4. TEST PROCEDURES

The recommended methods of test for compatibility of materials with liquid oxygen under impact are presented in the Appendix of this report. The document comprises, in essence, a description of test equipment and instrumentation as standardized, a step-by-step outline of an impact sensitivity

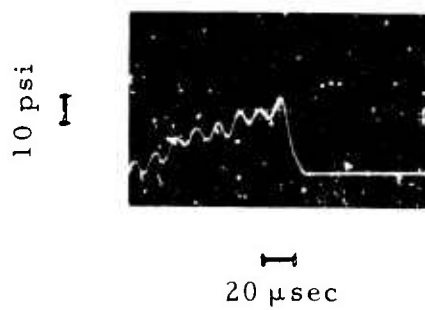


FIGURE 19. TRACE OBTAINED USING CUTOFF FILTER

test in terms of the 50-percent point, and a step-by-step outline of a reaction intensity test in terms of the maximum shock wave peak pressure.

To avoid confusion, the ABMA-type drop-weight apparatus complete with the selected mechanical and electronic components, as described in the Appendix, will be referred to as the AFAPL impact tester.

SECTION IV

RESULTS OF STANDARDIZED TESTS AND DISCUSSION

1. GENERAL

The results reported in this section were obtained in strict accordance with the equipment and procedures specified in the Appendix of this report.

It will be recalled from previous discussions that important differences exist between the responses exhibited by liquids and greases and those characteristic of solids. In essence, it appears that liquids and greases cannot be separated with reasonable confidence by impact sensitivity, while solids can be so separated. At the same time, reaction intensity ratings appear to be useful for both types of materials. Accordingly, the following discussions will take up each type of material separately, and it should be remembered that the conclusions drawn concerning one type do not necessarily apply to the other.

Further, it is pointed out that the reaction intensity measurements in this section cannot be directly compared to those on similar materials in preceding sections because of intervening equipment and procedure changes. For example, each individual test drop now yields two peak pressure readings, one from each of the two shock wave probes. As will be seen from the Appendix, the peak pressure for an individual test drop is defined as the higher of the two readings taken from the two peak pressure meters, and the maximum peak pressure for a complete test is defined as the highest of all peak pressure readings from twenty test drops or, in effect, the highest of forty readings. Thus, the average peak pressures and particularly the maximum peak pressures reported in this section will tend to be higher than those given in previous sections of this report.

2. RESULTS ON LIQUIDS AND GREASES

The presentation of the various data gathered on liquids and greases will be divided into five parts. The first part will compare the reaction intensity ratings of twenty-three different materials ranging from a chloro-fluorocarbon to a straight mineral oil. These ratings will demonstrate the test's ability to identify potentially hazardous materials. The second part will present the three types of peak pressure distributions found in the first investigation, with an attendant discussion of the implied effects of sample cracks and voids on the propagation process. The third part will present the results of reaction intensity tests on various mixtures of inert and explosive liquids. These data will show that it is very doubtful that a normally inert material can be made to react violently by the presence of a small amount of contaminant. The fourth part will compare the reaction

intensities of seven different batches of a commercial thread sealant. These and other data will be used to demonstrate that there is probably no significant change in batch-to-batch sensitivity of materials. The fifth part will present the evidence calling for a retesting of all liquids and greases currently in service.

a. Reaction Intensity Ratings for Twenty-Three Materials

It will be recalled from the preceding sections that the reaction intensity test by itself appeared to be capable of providing adequate information on the potential hazard associated with a liquid or grease. The object of the test series discussed below was to determine just how well this test, with the new intensity measuring equipment, would perform on a wide range of materials. As will be seen, it performed well indeed.

Figure 20 presents a comparison of maximum peak pressures obtained in sixty-nine different 20-drop tests on twenty-three different materials. For each material, the two circles connected by a line show the range of maximum peak pressures obtained in all tests on that material. For example, one 20-drop test on H-1091 yielded a maximum peak pressure of 50 psi, another test gave 32 psi, and each of the remaining eight tests yielded maximum peak pressures somewhere between these two values. Figure 21 is a copy of Figure 20 with the average peak pressure ranges included. It is, of course, immediately apparent that the best separation of materials is achieved by viewing the maximum peak pressure alone.

Before proceeding further with the discussion of these data, it is felt that some physical frame of reference for the various levels of peak pressure would be helpful. First, the maximum peak pressure readings obtained in two 20-drop tests on "blanks" (specimen cup with liquid oxygen but without any sample material) are included in Figure 20. These readings amount to background noise--the sound pressure generated by the steel-on-steel impact. Second, the readings from 0.4 to 1.2 psi (nine materials) are those of very faint sounds and are usually not accompanied by any visible flash. Especially toward the lower part of this range, it is often difficult for the operator to decide whether he has heard any sound of reaction. Third, somewhere in the range between 1.0 and 2.0 psi, reactions become definitely apparent, with small flashes and faint but distinctly audible reports. From about 2.0 psi up, reactions become more and more intense until finally, from about 20 psi up (fourteen materials), one can begin to see the power of the explosions by viewing the damage to the specimen cup sleeve. In the range from about 30 to about 50 psi, the 0.065-in. thick aluminum alloy sleeve (Fig. 15) was generally "barreled" or expanded in diameter by as much as 0.10 in. by the partially confined blast wave. From about 50 to about 90 psi, the sleeve usually expanded and split open. From about 90 psi up, the sleeve frequently fragmented.

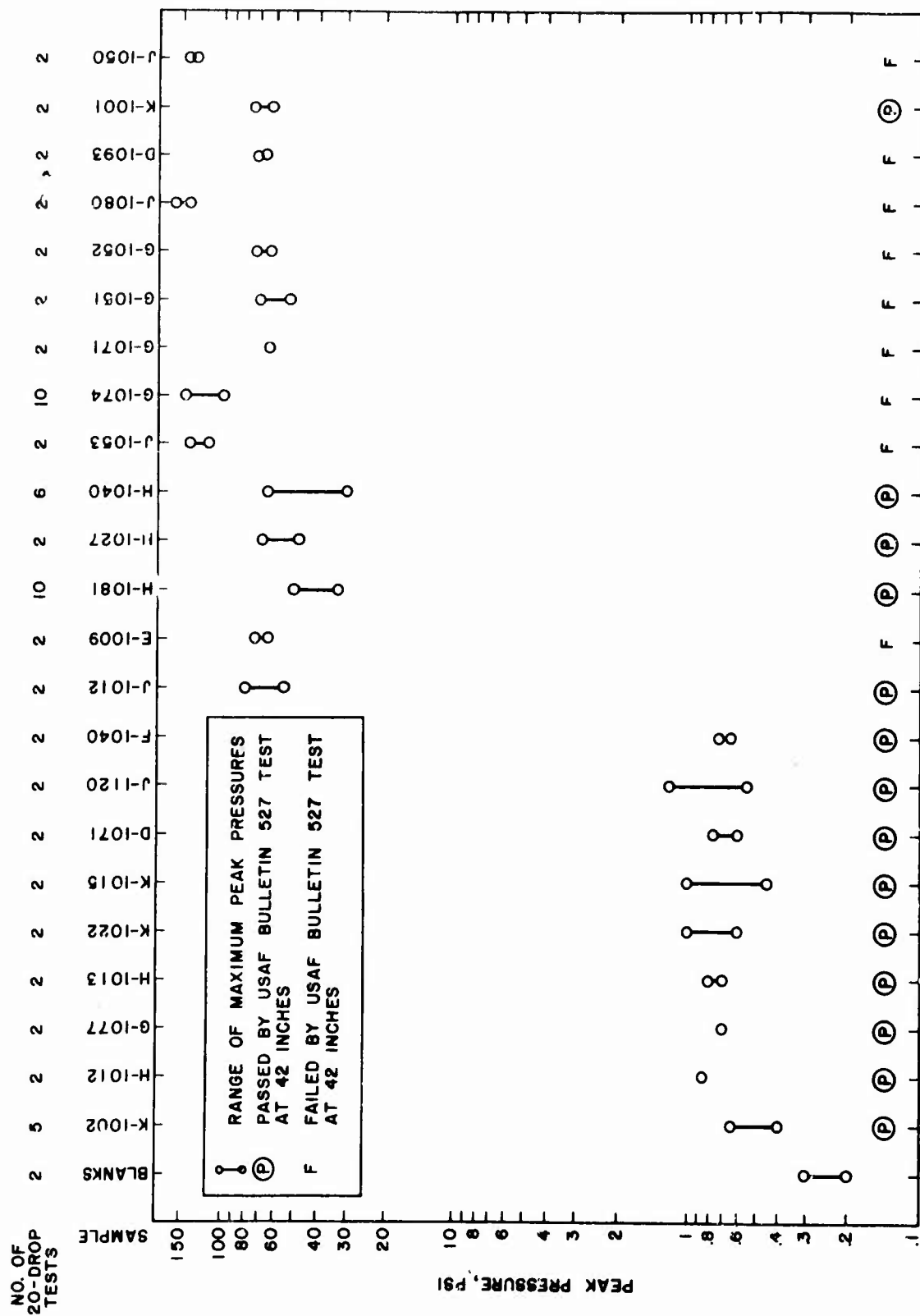


FIGURE 20. MAXIMUM PEAK PRESSURES OF VARIOUS LIQUIDS AND GREASES

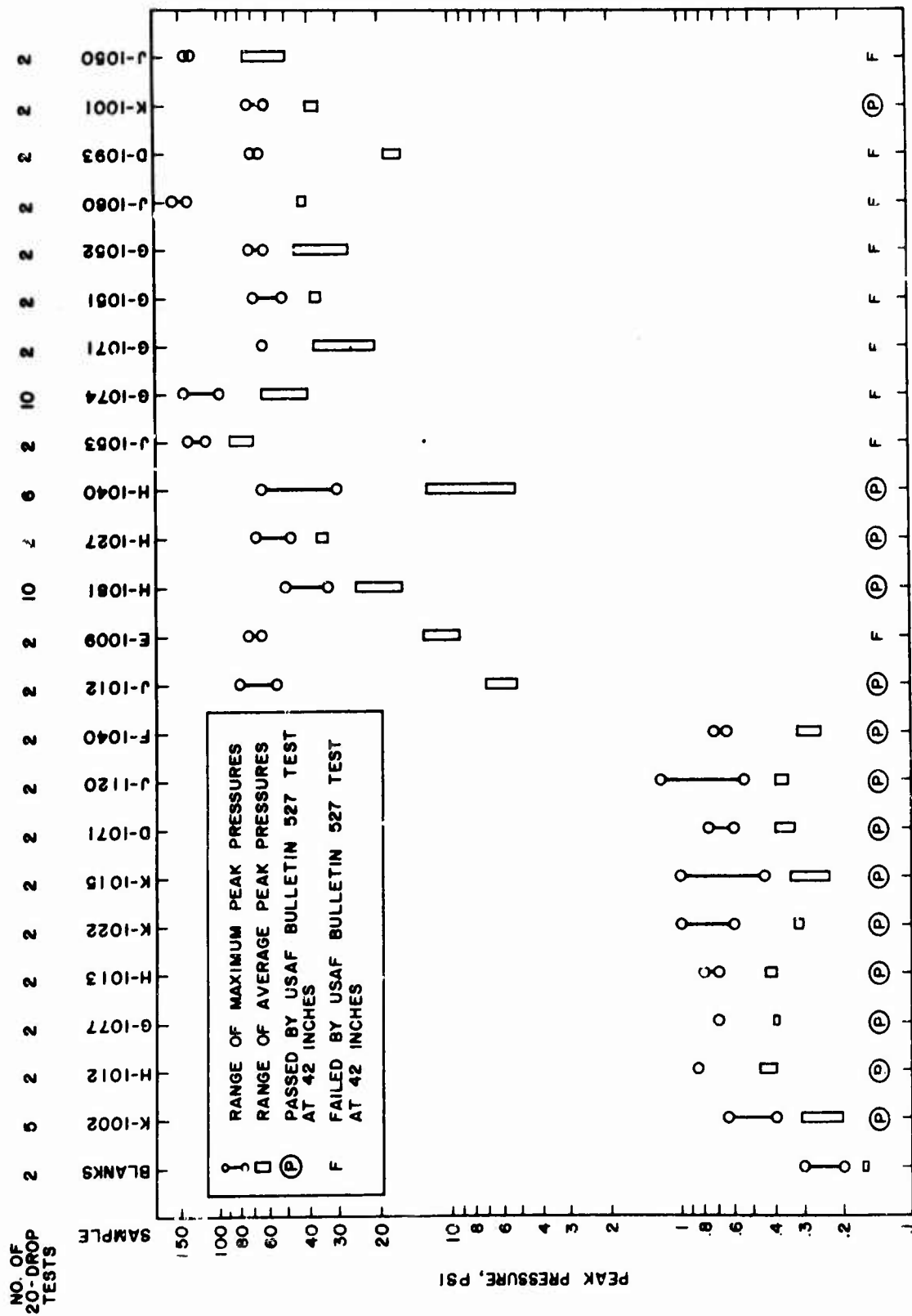


FIGURE 21. MAXIMUM AND AVERAGE PEAK PRESSURES OF VARIOUS LIQUIDS AND GREASES

Another physical reference which might be helpful would be the peak pressure readings generated by a form of explosive with which many are familiar--the firecracker. Such data were obtained on both "Chinese firecrackers" and "cherry bombs." The "Chinese firecracker," or flash-cracker, is the type of small 1-1/2-in. long firecracker which comes tied together in strings of twelve or more, so that lighting of the main fuse causes sequential ignition of the entire string. For this test, ten of these firecrackers were detached from the string, placed one at a time in the specimen cup, and ignited with matches. For these ten, the average peak pressure was 7.0 psi, and the maximum peak pressure was 8.6 psi. Ten "cherry bombs" (the spherical firecracker of about 1.0-in. diameter) were tested similarly, yielding an average peak pressure of 58 psi and a maximum peak pressure of 70 psi. Several of these latter explosions were powerful enough to split the sleeve, as were explosions of similar magnitude in Figure 20. It should also be noted that several materials in Figure 20 yielded explosions considerably more powerful than a "cherry bomb."

Before leaving the discussion of physical references, it should be emphasized that the range of 1.0 to 2.0 psi, in which reactions become definitely apparent, is rather broad. Furthermore, less definitely apparent evidence of reaction, such as a tiny char mark, is sometimes found even though the peak pressure reading might be as low as 0.2 psi. Then again, the same peak pressure reading might be accompanied by no evidence of reaction whatever. From this, it can be seen that peak pressure measurements alone cannot always reveal whether a reaction did or did not occur; their usefulness is restricted to the comparison of intensities.

It is evident from Figure 20 that the twenty-three widely different materials tested fell neatly into two very distinct groups: one with high maximum peak pressures and the other with low maximum peak pressures. Judging from the physical references discussed above, it is not unreasonable to assume that a "30-psi" reaction would probably abort the mission of an oxidizer system as effectively as one with 160-psi maximum peak pressure. On the other hand, reactions with less than 1.2-psi maximum peak pressures were generally so mild that system damage would not appear likely. Viewed in this light, it can be seen that one of the groups of materials in Figure 20 could be considered extremely hazardous and the other relatively inert.

Note should also be made of the fact that at least two 20-drop tests were made for each material shown in Figure 20. The results show that the range of variation of the maximum peak pressures from the different 20-drop tests on each material were generally in excellent agreement, particularly with respect to classifying whether the material was extremely hazardous or relatively inert. There was no instance in which the material would be considered extremely hazardous in one 20-drop test and relatively inert in another. In other words, it appears that a single 20-drop test would be quite adequate for rating the relative hazard of materials in terms of

maximum peak pressure. This is because a 20-drop test yields, in reality, 40 peak pressure readings, a number apparently sufficient to give a representative maximum peak pressure value with a high degree of statistical confidence.

Also worthy of mention is the fact that no less than five of the materials shown to be capable of powerful explosions in Figure 20 have previously passed the Bulletin 527 test at 42 inches. These are J-1012, H-1081, H-1027, H-1040, and K-1001.

As seen above, the maximum peak pressure obtained in a 20-drop test seems to give sufficient information for rating purposes. However, referring now to Figure 21, there are some interesting factors about the average peak pressures exhibited by these twenty-three materials. It will be noted that, in the majority of cases, the average peak pressure was not far removed from the maximum peak pressure. On the other hand, three materials--E-1009, J-1012, and H-1040--exhibited average peak pressures significantly lower than their maximum peak pressures. In addition, of the nine materials back in Figure 14, J-1012 and H-1040 were the only two demonstrating substantially higher peak pressures in Figure 20. These behaviors are believed to be the result of erratic reaction propagation, as will be seen in the following discussion.

b. Typical Peak Pressure Distributions

Three significantly different types of peak pressure distributions were found in the testing of the materials included in Figure 20. As will be seen, one type in particular suggested a possible problem concerning the number of test drops required in a single reaction intensity test.

Figure 22 is a bar graph of the numbers of peak pressure readings falling within selected ranges for H-1081. Note that lines on the graph enclose the "readable peak pressure scale." This refers to a limitation caused by the fact that the peak pressure meter readout scale is confined to readings from 0.1 to 10.0 volts, so that the maximum range on any selected charge amplifier range setting is 100 units. Thus, if it is desired to measure peak pressures up to 100 psi, then any peak pressures of less than 1.0 psi will not register. In this case, the limited range was of no consequence since all 200 test drops produced readings in the range from 7 to 50 psi. This rather narrow and relatively symmetrical distribution of peak pressure was also observed for all nine relatively inert materials as well as the more explosive materials, H-1027, J-1053, G-1071, G-1051, G-1052, and K-1001. Interestingly, all the explosive materials in Figure 20 in this category were greases.

The second type of peak pressure distribution is typified by G-1074 in Figure 23. Of the 200 test drops on this material, 171 produced

THE LENGTH OF EACH BAR REPRESENTS THE NUMBER
OF READINGS FALLING WITHIN THE VALUES ENCLOSED
BY THE WIDTH OF THE BAR

THE PEAK PRESSURE SCALE WAS 1.0 TO 100 PSI

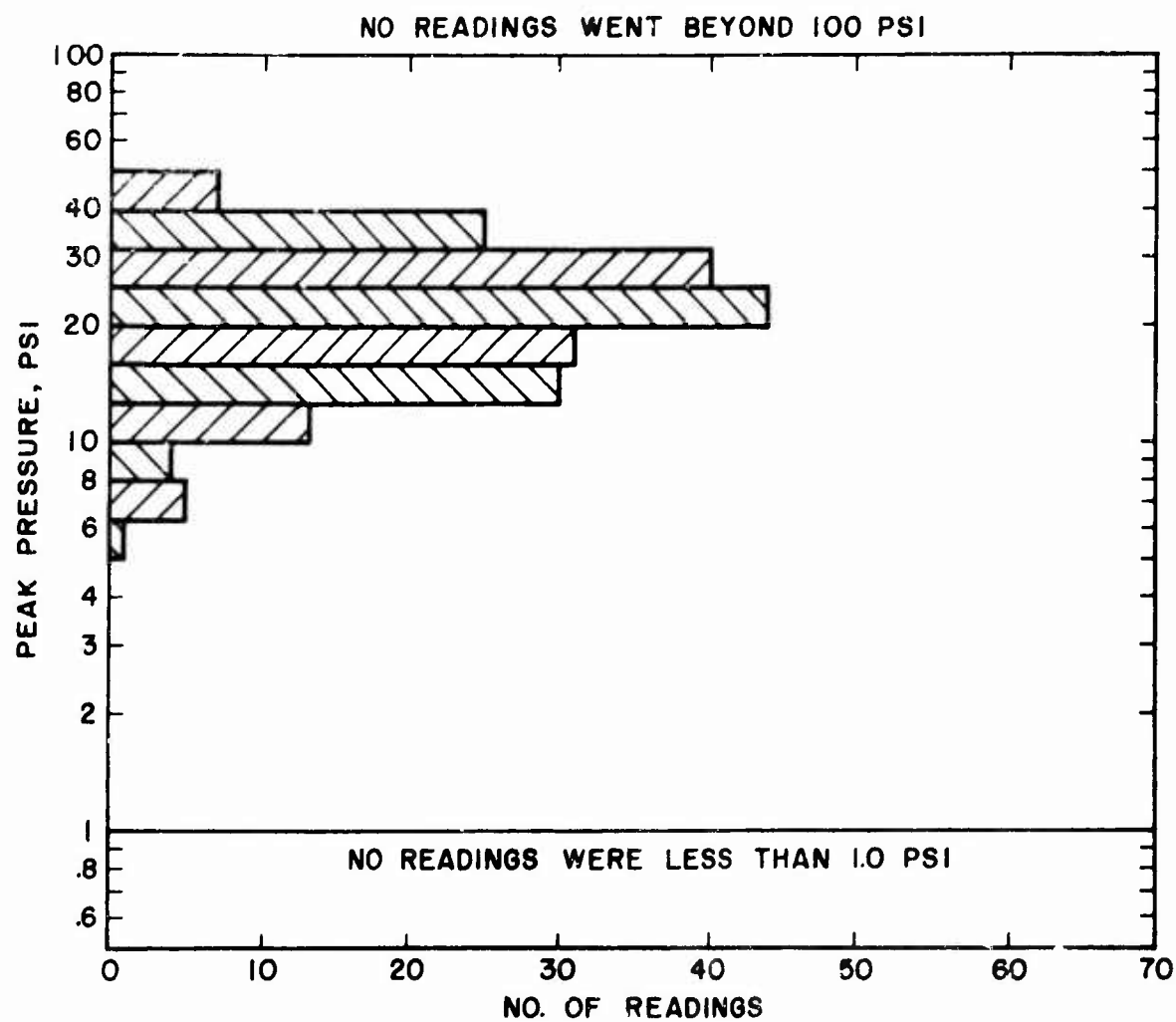


FIGURE 22. DISTRIBUTION OF PEAK PRESSURES FOR H-1081

THE LENGTH OF EACH BAR REPRESENTS THE NUMBER
OF READINGS FALLING WITHIN THE VALUES ENCLOSED
BY THE WIDTH OF THE BAR

THE PEAK PRESSURE SCALE WAS 20 TO 200 PSI

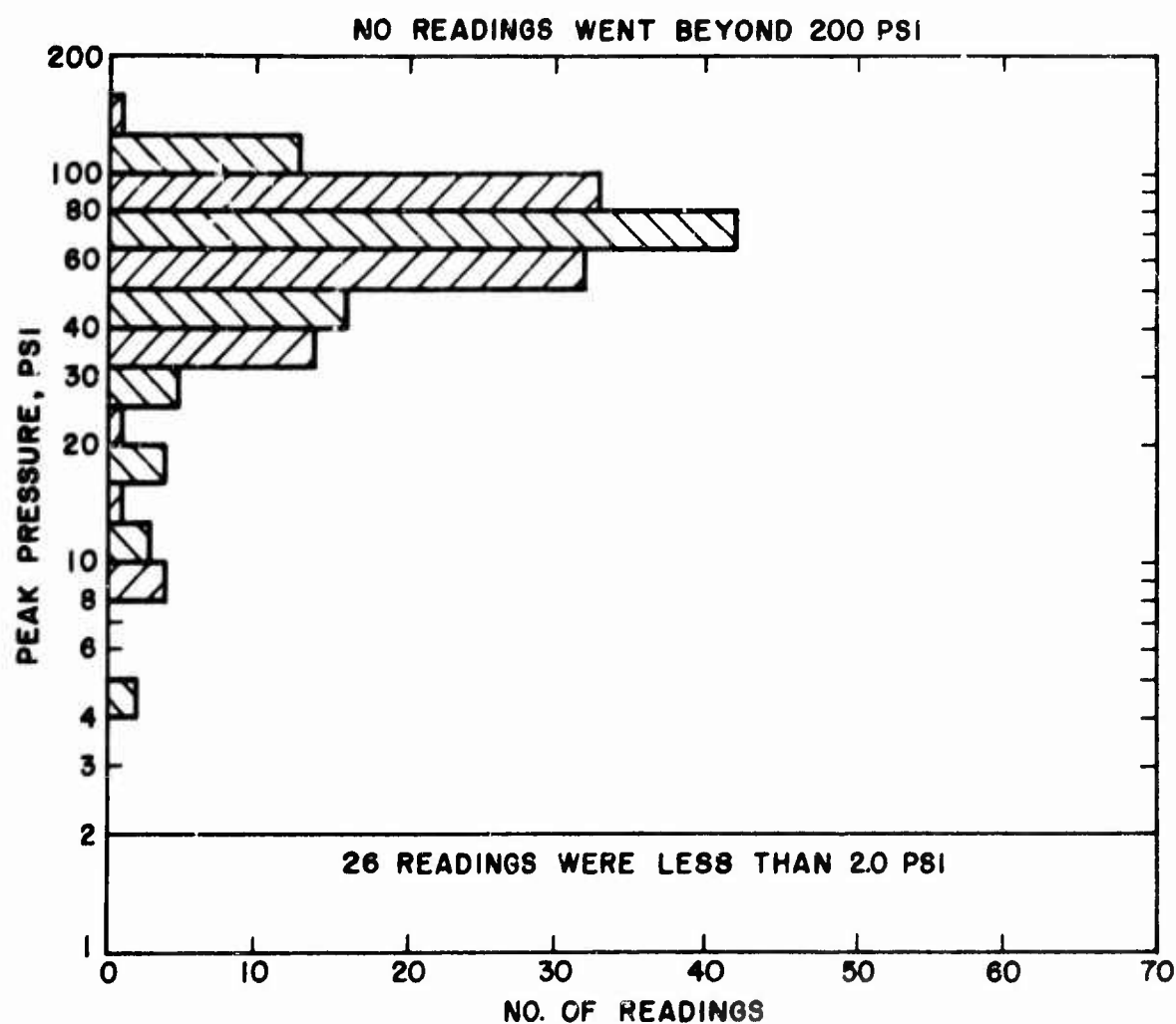


FIGURE 23. DISTRIBUTION OF PEAK PRESSURES FOR G-1074

readings in the readable range of 2.0 to 200 psi, twenty-six produced no readings but showed evident char, and three gave neither readings nor any other evidence of reaction. Note that the erratic behavior here was not in initiation (99 percent of the test drops initiated reactions), but in propagation since only 86 percent of the test drops produced reactions propagating past 2 psi. This is in contrast with the results above on H-1081, in which every initiation propagated past 2 psi. Other explosive materials in Figure 21 showing a skewed distribution similar to G-1074 were J-1080, D-1093, and J-1050. Note that all four are liquids.

The third type of peak pressure distribution is demonstrated by H-1040 in Figure 24. Of the 120 test drops on this grease, fifty-one propagated past 1.0 psi, forty-eight produced no reading but evidence of reaction, and twenty-one produced no sign of reaction. Again, the erratic behavior was not so much in reaction frequency, since 82 percent of the test drops initiated reactions, as in propagation since 42 percent of the test drops produced propagating reactions. Exhibiting this rather broad but flat peak pressure distribution were, in addition to H-1040, J-1012 (a grease believed to be of similar constituency to H-1040) and E-1009 (a liquid believed to be a component of J-1012 and H-1040). All three are explosive materials.

In considering these three types of propagation behavior and certain physical differences in the types of material, a pattern appears which may have a bearing on the efficiency of the impact test. Since considerably more cracking takes place in a frozen liquid sample than in a frozen grease sample, and since the liquids show many more propagation failures than the greases, it appears that a propagating reaction can sometimes lose its momentum and die out, so to speak, when it encounters a crack. Further, the two greases in the third category, while cracking less than the liquids, tended to "float" somewhat in spite of the best efforts to prevent them from doing so. The liquid in the third category also tended to "float." Thus, the cracking dislocations were probably enhanced and further voids created by "floating" in the third category which, in turn, apparently further depressed the frequency of propagating reactions. Before proceeding further, it should be mentioned that one of the greases in the first category, J-1053, is also a "floater" but had no propagation failures. This could be accounted for by assuming that this material's very high reaction rate reduced cracks and voids to mere minor impediments.

In an attempt to verify experimentally the above-assumed effects of "floating," 120 test drops were made on H-1040 samples which were caused purposefully to "float" much more than those of the previous tests. In this second series, only sixteen reactions propagated to 1.0 psi or higher, considerably less than the fifty-one obtained in the first series. Thus, it can be seen that "floating" of the sample material apparently does tend to retard reaction propagation. Furthermore, the results in Figure 20 for both J-1012 and H-1040 were obtained on samples with which special care was taken to

THE LENGTH OF EACH BAR REPRESENTS THE NUMBER
OF READINGS FALLING WITHIN THE VALUES ENCLOSED
BY THE WIDTH OF THE BAR

THE PEAK PRESSURE SCALE WAS 1.0 TO 100 PSI

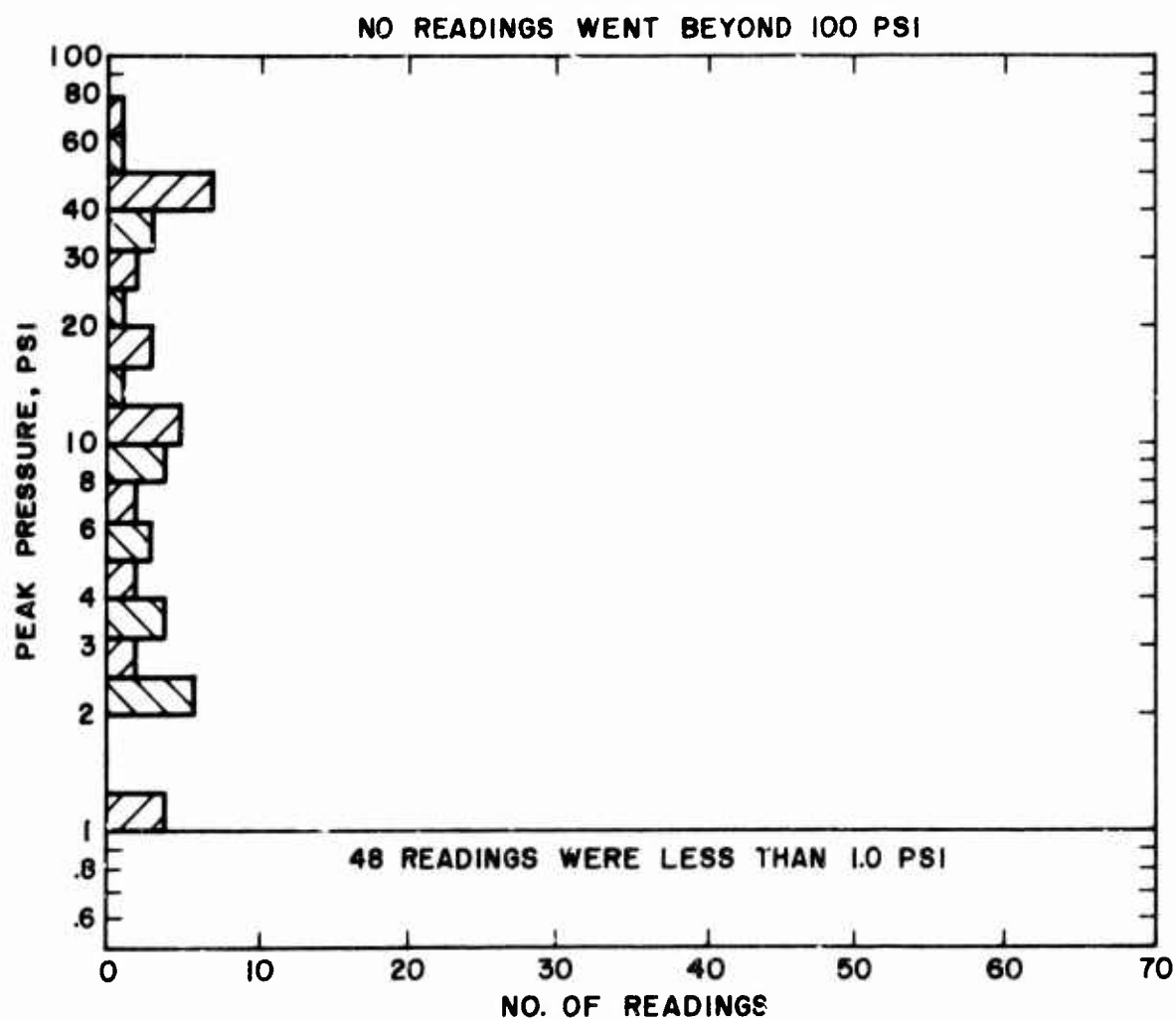


FIGURE 24. DISTRIBUTION OF PEAK PRESSURES FOR H-1040

prevent "floating." This, together with the differences in peak pressure measuring equipment and technique, is believed to have contributed to the different behaviors of these two materials in Figures 14 and 20.

Now, to get to the possible influence of sample "floating" on the reaction intensity test efficiency, first recall that this test must determine in a limited number of drops whether a material can produce an explosive reaction. In the case of the "floated" H-1040 samples above, which had 13-percent propagation, reference to a table of cumulative binomial probabilities⁽¹³⁾ reveals that a 20-drop test would not produce a propagating reaction about 6 percent of the time. If that 13 percent above were reduced to, say, 5 percent, the chances of not having a propagating reaction in a 20-drop test would rise to about 35 percent. In other words, there would be significant danger that the test would fail to identify a potentially hazardous material. Therefore, two recommendations concerning material qualification with the reaction intensity test must be made. First, all reasonable care, such as very slow freezing and gentle handling, should be taken to minimize floating. Second, if a candidate material tends to "float" in spite of all measures, then it should be required to survive many more test drops (say, sixty or more) without producing a propagating reaction.

Fortunately, experience indicates that the above large number of test drops will very rarely be necessary since most of the materials tested that do not propagate have shown little tendency to "float." On the other hand, most materials capable of producing violent reactions will do so on the first or second test drop most of the time, eliminating the need for further drops.

c. Further Tests on Liquid Mixtures

It will be recalled that, during the period of development of the reaction intensity measuring system, reaction intensity measurements at 43.3 in. were made on various mixtures of E-1010 and J-1080 (Table 9). Considering the intervening equipment changes, it was decided to bring these measurements up to date. Table 14 presents the newly obtained reaction intensity test results on mixtures of K-1081 and K-1082 (different batches of the same liquids).

In considering these results, it should be recalled from Section IV.2.a that a reaction becomes definitely apparent at a peak pressure somewhere between 1.0 and 2.0 psi. With this in mind, it can be seen that a definitely apparent reaction did not occur until the proportion of the explosive liquid (K-1081) was increased to 20 percent. Even then, only one such reaction, and it was of low order, occurred in five 20-drop tests. Further, it was necessary to increase the proportion of the explosive liquid to 70 percent before highly explosive reactions became numerous.

TABLE 14. REACTION INTENSITIES OF MIXTURES
OF TWO LIQUIDS

<u>Sample</u>	<u>Avg. Peak Press., psi</u>	<u>Max. Peak Press., psi</u>
K-1082	0.18	0.36
90% K-1082 - 10% K-1081	0.20	0.54
	0.27	0.65
	0.21	0.40
	0.27	0.65
	0.24	0.40
80% K-1082 - 20% K-1081	0.17	0.25
	0.25	0.35
	0.24	0.55
	0.22	0.75
	0.48	2.25
70% K-1082 - 30% K-1081	0.49	6.8
	0.18	2.5
	0.23	1.5
	0.35	5.5
	1.33	21.0
50% K-1082 - 50% K-1081	6.3	46.0
30% K-1082 - 70% K-1081	38.0	100.0
K-1081	48.0	124.0

Note: Each average and maximum peak pressure comes from a 20-drop test at 43.3 inches.

Of course, there is enough variance in these results that one could not determine precisely the maximum proportion of explosive liquid allowable without danger of explosion. However, it is apparent that proportions of 10 percent or less would appear to be safe in terms of explosion hazard. Now, again, this is not to say that initiations do not occur in the lower proportions of the explosive liquid, but rather that the reactions do not seem to be able to propagate enough to be dangerous.

The above suggests that a revision in current thinking about the "contamination" problem may be in order. It has been generally accepted that minute quantities of some contaminants can render an inert material impact sensitive. Now, if one accepts the premise of the rather large volume of systematic data presented in this report, the term "impact sensitive" becomes quite irrelevant since it has been shown that the sensitivities of most liquids and greases are about the same regardless of whether they are relatively inert or highly reactive. On the other hand, the criteria that is relevant--reaction intensity--suggests that as much as 10 percent contaminant can be present without danger of an explosive reaction. Of course, these arguments are not intended to imply that reasonable efforts to avoid contamination of liquids and greases are not needed; rather, they are presented to emphasize that an explosive reaction obtained with a supposedly inert material in a test or service condition cannot necessarily be blamed on the presence of trace quantities of some other highly reactive material.

d. Relative Ratings of Seven Different Batches of a Grease

In a previous report⁽¹¹⁾, 50-percent points were determined for seven different batches of a commercial thread sealant. These batches were submitted by AFAPL, and it was understood that all seven had been rated as satisfactory in the Bulletin 527 test at various other laboratories. The 50-percent points obtained ranged from 9.4 to 12.0 in., and it was concluded that no significant sensitivity differences existed for any of the seven.

To add to the information on these batches, all seven were subjected to 20-drop reaction intensity tests. The results are presented in Table 15. In considering these results, it is apparent that this test says that all seven were unsatisfactory, with each demonstrating its explosive capability.

The above discussion brings to mind another generally accepted conclusion about material behavior--that the sensitivity of certain liquids or greases varies from batch to batch. Although SwRI has not been able to obtain the same material in different batches that have both passed and failed the Bulletin 527 test at 42 in., the following considerations cast serious doubt upon the validity of this conclusion. First, as discussed in a previous section, sensitivity differences shown by the Bulletin 527 type test are probably due more to the test method than to any characteristic of the material being tested, and this, by itself, casts considerable doubt upon such

TABLE 15. REACTION INTENSITIES OF SEVEN DIFFERENT
BATCHES OF A COMMERCIAL THREAD SEALANT

<u>Sample</u>	<u>Avg. Peak Press., psi</u>	<u>Max. Peak Press., psi</u>
J-1013	2.7	40.0
J-1014	6.7	32.0
J-1015	6.9	38.0
J-1016	8.2	43.0
J-1017	10.1	36.0
J-1018	11.3	58.0
J-1019	4.9	37.0

Note: Each average and maximum peak pressure comes from a 20-drop
test at 43.3 inches.

conclusions drawn from data obtained using the Bulletin 527 test. Second, at SwRI, a single batch of H-1081 has yielded both passes and failures in 20-drop tests at 42 in. using the Bulletin 527 method--further proof that the method itself varies from test to test. Third, both H-1081 and the material tested in seven batches above are shown to be capable of explosion, as does E-1009, another material presently thought to vary in sensitivity by batch. Considering all the evidence, it appears probable that all batches of materials whose sensitivity is thought to vary are actually all explosive and that the Bulletin 527 test results are merely a sporadic indication of this.

e. The Need for Retesting All Liquids and Greases Currently in Service

It has been pointed out several times previously that several apparently hazardous liquids and greases are capable of passing the Bulletin 527 test in twenty drops at 42 in. without reaction. Considering that, for the past several years, this test method and its very similar NASA counterpart have handled nearly all the testing in this area, it is a logical assumption that such hazardous materials may inadvertently be in service at present. Therefore, it is strongly recommended that all liquids and greases currently in service (or being considered for service), by virtue of having passed one of these test methods, be retested with the reaction intensity test.

Admittedly, this recommendation may appear at first to be disturbing from a practical standpoint. Over the years of use of the two threshold value test methods, hundreds of thousands of test drops have been made, and it can be argued that a change in the test method would nullify all the results of this vast amount of work. Actually, it should be pointed out that the recommendation as stated applies only to a rather limited number of liquids and greases. By far the greater part of the past work was done on materials which at once demonstrated their potential hazard (by exploding) with the threshold value test methods. Such results cannot be nullified, and there appears to be little need for retesting such materials.

3. RESULTS ON SOLIDS

It will be recalled from previous discussion that both impact sensitivity and reaction intensity may be necessary to the evaluation of solids. Considering this, two test series were conducted in this investigation. The first test series involved sensitivity tests on various thicknesses of several solids using four different striker pin diameters. The second test series was comprised simply of reaction intensity tests from 43.3 in. on these and other materials. After the discussions of these data, there will be comments on the service history of Mylar film compared with its performance in these tests.

a. Impact Sensitivity Tests

Figures 25 through 28 present the sensitivities obtained for Mylar (Type A), acetate, Kapton, and phenolic with both sample thickness

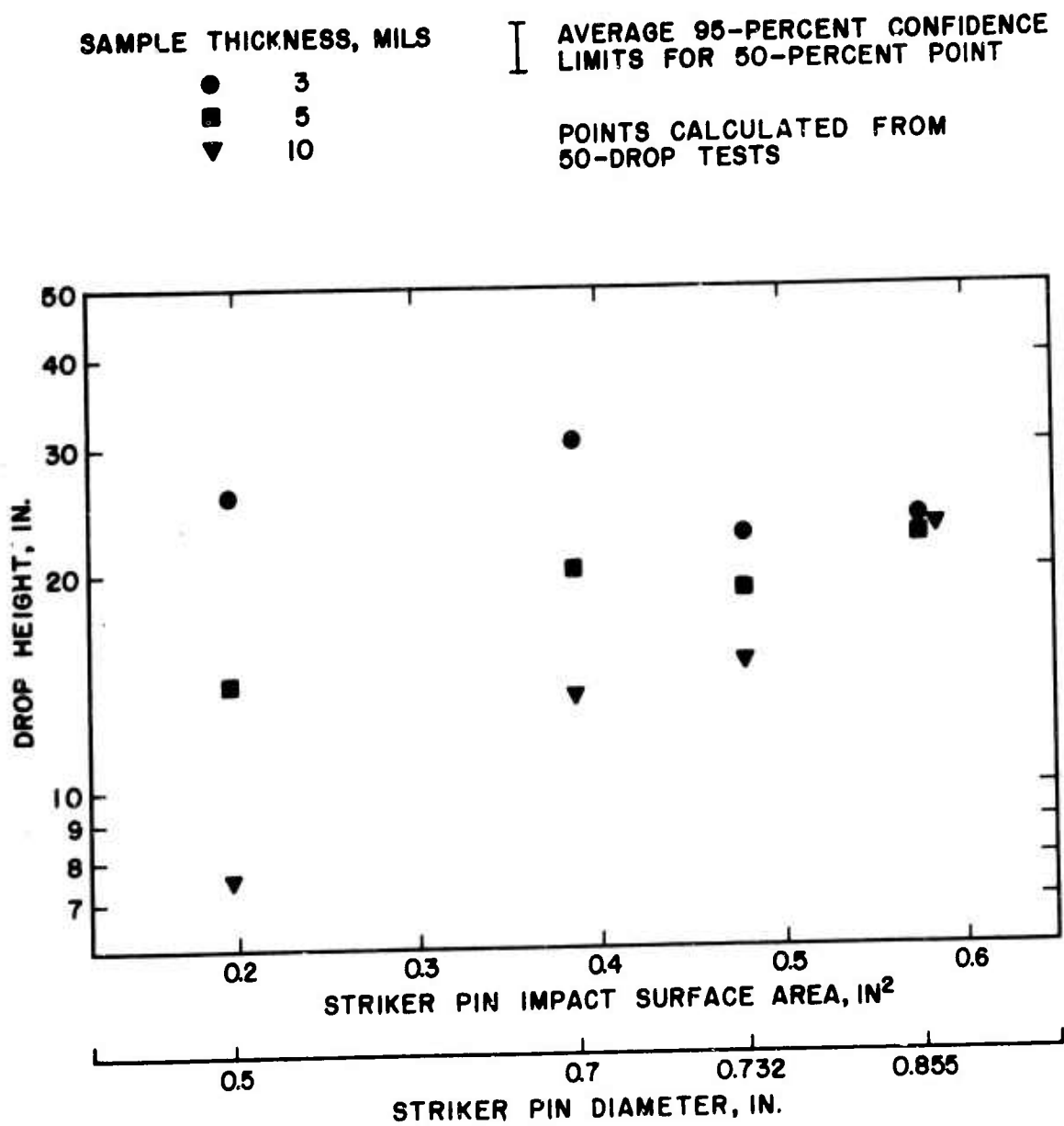


FIGURE 25. 50-PERCENT POINT VS SAMPLE THICKNESS AND IMPACTED AREA FOR MYLAR, TYPE A

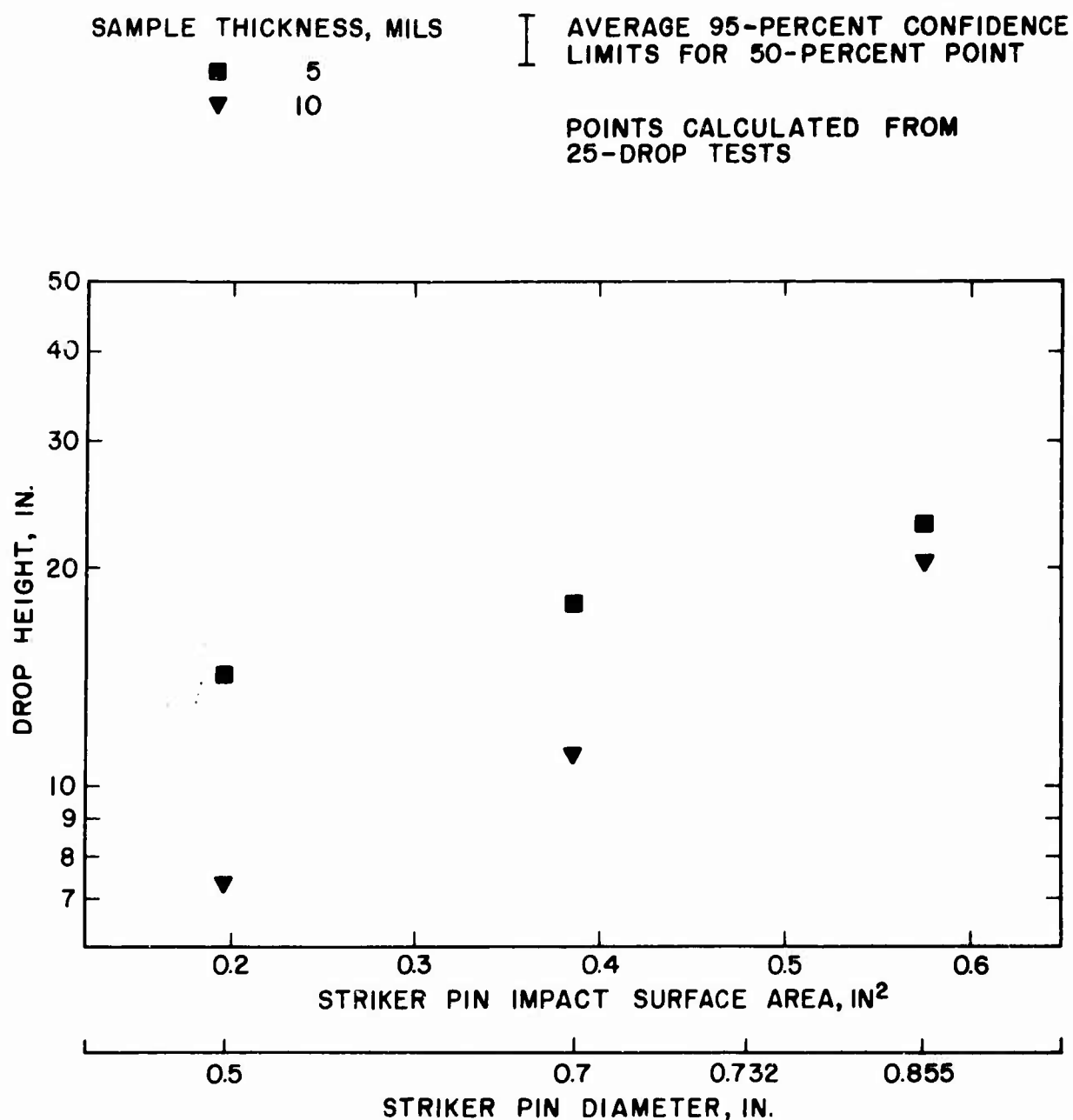


FIGURE 26. 50-PERCENT POINT VS SAMPLE THICKNESS
AND IMPACTED AREA FOR ACETATE

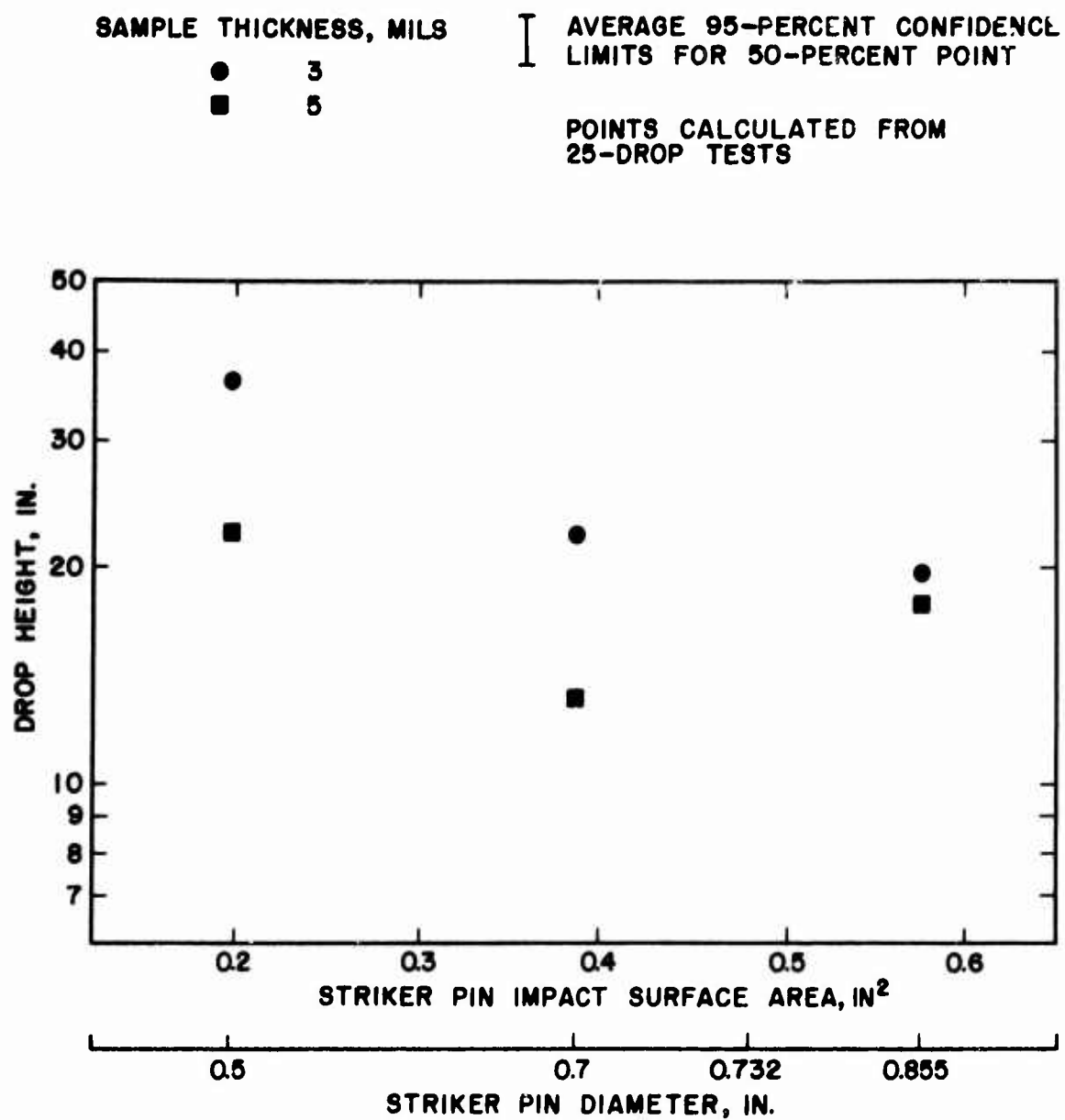


FIGURE 27. 50-PERCENT POINT VS SAMPLE THICKNESS AND IMPACTED AREA FOR KAPTON

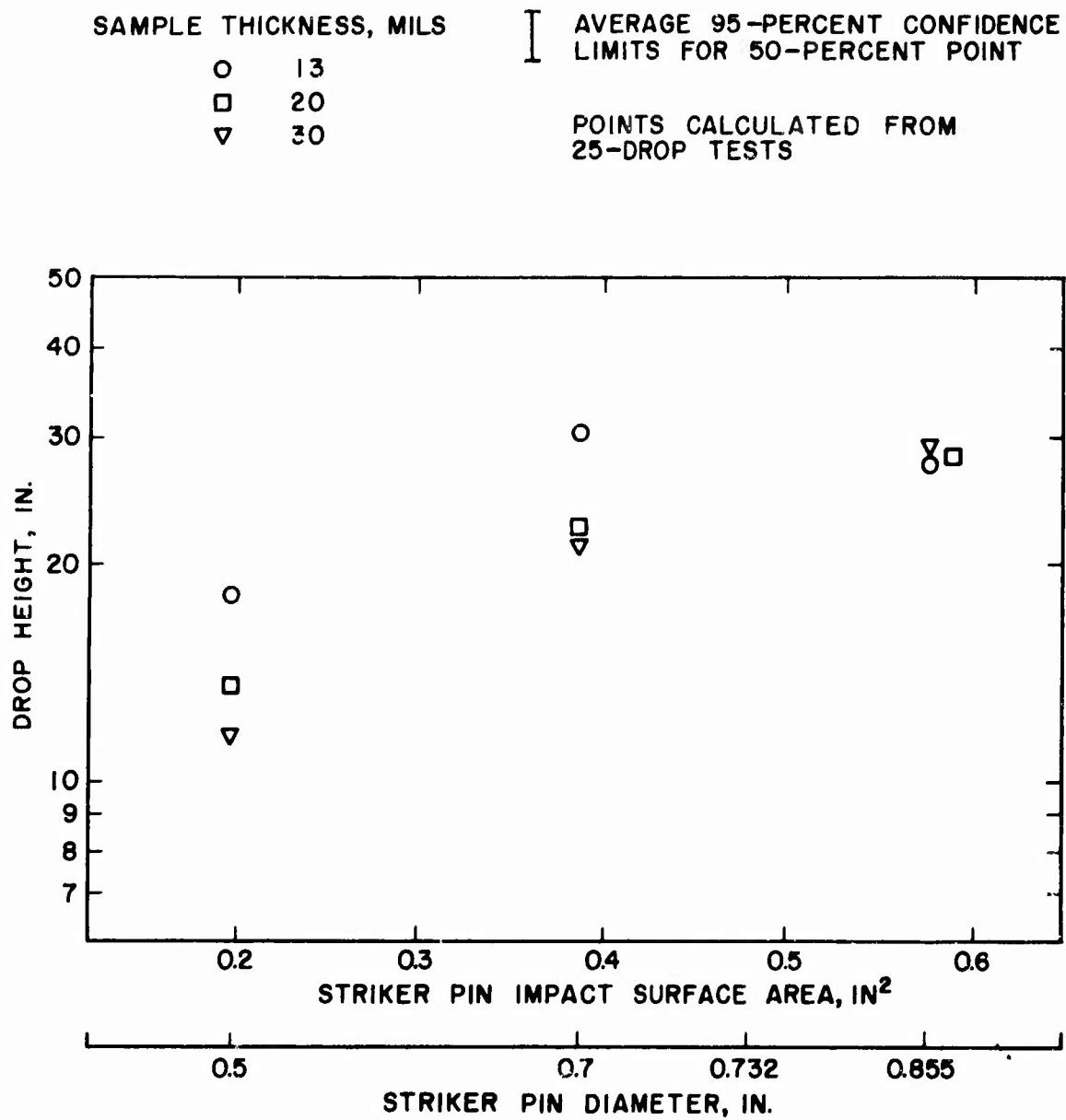


FIGURE 28. 50-PERCENT POINT VS SAMPLE THICKNESS
AND IMPACTED AREA FOR PHENOLIC

and striker pin impact surface area as variables. In each graph, the ordinate is drop height on a logarithmic scale, and the abscissa is striker pin impact surface area on a linear scale. Also, in each graph is a scale of the typical 95-percent confidence limits for the 50-percent points for that graph (the probability that the population mean lies within these limits is about 95 percent). By visually placing the scale astride the various 50-percent points, one can see at a glance which points are significantly separated. These scales are no more than approximations since each was computed from the average of the standard deviations in its own graph; however, the spread of the standard deviations was small (for solids), and these scales are quite adequate for the intended usage. Simultaneous consideration of these four graphs reveals several interesting trends which will be discussed.

Considering sample thickness, it will be noted that the widest separation of sensitivities was shown with the 0.5-in. diameter striker pin. This separation decreased with increasing impact area until, with the 0.855-in. diameter striker pin, there was no significant separation. Generally, it is seen that, for all four materials, the thinnest samples were the least sensitive, and the thickest were the most sensitive. This appears to be just the opposite of what one might expect, and, indeed, there is no readily apparent explanation for this behavior. The evidence, however, is undeniable that sample thickness is a major factor.

Considering impact surface area, it is seen that, with the exception of both Kapton samples and the 3-mil Mylar, the materials tended to show less sensitivity as the striker pin diameter increased. This was not surprising since increasing the striker pin impact surface area in effect diluted the impact energy delivered. On the other hand, the exceptions to this behavior show clearly that impact energy delivered to a given area is not the only influence on the sensitivity of solids, even with sample thickness held constant.

Further evidence of the various reaction mechanisms involved in these tests can be seen in the tendency of the sensitivities of the various thicknesses of all four materials to converge as striker pin area is increased. Apparently, the influence of the reaction mechanism associated with sample thickness can be completely overshadowed by other influences such as striker pin area or sample entrapment.

From the above, it can be seen that a great deal of information about the sensitivity behavior of various solids can be gathered through the simple expedient of varying the striker pin diameter. Also, it should be noted that all this information came from only thirty-two data points. Moreover, the tests with the 0.782-in. diameter striker pin in Figure 25 could have been omitted without significant loss of information, and the other tests in this graph could have been limited to twenty-five drops without significant loss of accuracy. Thus, the various trends revealed here would have been just as evident with only twenty-nine tests of twenty-five drops each. Since this is a matter of only about six working days, it can be seen that this test is quite efficient.

b. Reaction Intensity Tests

Figure 29 presents the reaction intensity test results on various thicknesses of eight different solids. Here, as with liquids and greases, the concern is with the potential hazard associated with the various materials, and the best measure of this is the maximum peak pressure.

It is readily apparent from Figure 29 that the reaction intensity test does not provide the same clearcut distinction between reactive and relatively inert solids that it did for liquids and greases. However, three fairly distinct groupings do appear:

- (1) Teflon;
- (2) Vinyl, all Mylars, all Kaptons, and 5-mil acetate; and
- (3) 10 and 20-mil acetates and all phenolics.

The lone material in the first group above, Teflon, has the unique distinction of being the only material of any type tested in this program that showed absolutely no evidence of reaction. Thus, the peak pressures shown may be attributed to background noise.

Another new type of behavior is shown by the materials in the second group. These materials exhibited peak pressures (and thus, audible sound) not much higher than the relatively inert liquids and greases in Figure 20. Yet, nearly all drops produced visible flashes and distinct charring, whereas the liquids and greases in question produced no flashes whatever and rather indistinct charring. Apparently, the propagation rate for these materials is fast enough to produce visible burning but too slow to produce a sharp air shock wave. In other words, these materials apparently are combustible but not explosive. Then, too, the burning dies out quickly even though the sample may not have been broken up by the impact, thus indicating a self-quenching nature.

The third group showed reaction intensities of an order that should be regarded as hazardous almost to the same degree as the highly explosive liquids and greases.

In the preceding section on impact sensitivity of solids, it was mentioned that sensitivity alone was not sufficient to qualify solids. Before proceeding with this discussion, it should be recalled that a sensitivity rating, which is concerned only with the occurrence of reaction, gives very little quantitative information about reaction propagation--the real determinant of whether service system damage can occur. Admittedly, propagation cannot occur without ignition; however, knowledge of the test parameters controlling ignition is useful only in a relative sense unless one knows the

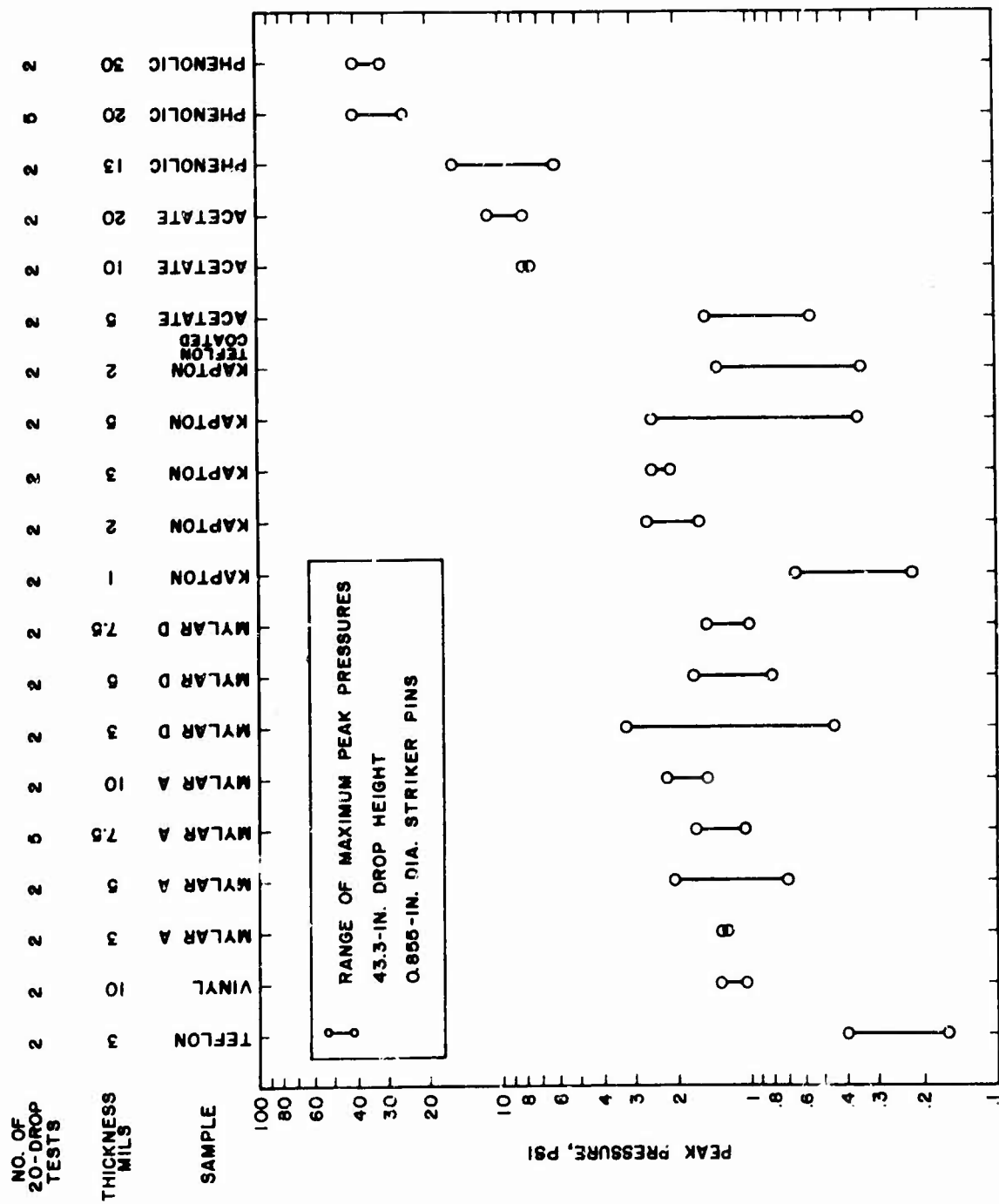


FIGURE 29. MAXIMUM PEAK PRESSURES OF SOLIDS

correlation between test and service parameters. On the other hand, a reaction intensity rating is more nearly absolute in the sense that a material exploding in the test has proven its potential for exploding in service. With these things in mind, it can be shown that sensitivity ratings alone might be misleading. As a hypothetical situation, assume that one was forced to choose between 10-mil Mylar and 10-mil acetate for some service application (no other material or material thickness would do) and that acetate was considerably cheaper than Mylar. With the sensitivities of these two being almost the same (Figs. 25 and 26), the economic factor might prompt the choice of the acetate. On the other hand, if one then considered the reaction intensity ratings, this choice would promptly be reversed since 10-mil acetate is in the explosive range, while 10-mil Mylar is only borderline in this aspect. Thus, it is seen that measurements of both parameters--reaction intensity and sensitivity--are necessary if informed choices are to be made.

There are, of course, exceptions to the general rule of making both measurements. For example, if a material having the degree of inertness shown by Teflon can be used, then only the reaction intensity test is needed. This becomes obvious when one considers that the sensitivity test requires reactions of some sort to produce a rating and that the simpler reaction intensity test would reveal no evidence of reaction in this case. Unfortunately, however, it does not appear that such inert materials will prove to be the best choice in every case. An example of this is the material to be used for oxidizer expulsion bladders, a requirement for which Mylar and Kapton are being considered because of their favorable low-temperature properties.

It was previously mentioned that knowledge of sensitivity parameters is useful only in a relative sense without some known correlation of test and service conditions. The subject of whether such a correlation is already within reach will be discussed in the following paragraphs.

c. Test Results vs Service History of Mylar

In selecting materials for diaphragm, lip seal, and flange gasket applications in rocket engine oxidizer systems, one of the major difficulties is that some of the polymeric materials most desirable from the physical-property standpoint will not pass present-day impact tests. On the other hand, one of these materials, Mylar film, is reported to have survived thousands of liquid oxygen service exposures without detrimental effect. Thus, it would seem that a reassessment of this material's service and test behavior is in order.

There is, of course, no question that Mylar reacts in the impact test as this is verified in Figure 29. The purported successful service history of Mylar must, therefore, be attributed to one or both of the following factors:

- (1) The service conditions are insufficient to cause reaction.
- (2) Reactions occur, but not of sufficient intensity to cause explosions.

The second factor above must be considered because of the relatively low reaction intensities shown by Mylar in Figure 29. If it is possible to calculate the service shock parameters seen by a solid (Section II. 7. b), then a determination of the parameters in effect while Mylar was in service might reveal which of the above factors was prevalent. For example, if the service shock parameters were considerably less than those required to produce the 50-percent points in Figure 25, then the first factor above could be considered prevalent. If not, then the second factor should be suspected.

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

1. CONCLUSIONS

Many investigators have long suspected that the poor repeatability and reproducibility of the existing impact sensitivity test methods are largely due to the statistical inadequacy of the threshold value concept. In this report, the best available data from the threshold value type test methods were compared with the data derived from a statistically more reliable 50-percent point test method, and the above suspicion was shown to be well justified.

Using the 50-percent point test method, it was shown that the impact sensitivities of many liquids and greases (in fact, nearly all of those tested) actually lay within a very narrow range (between 10 and 20 in.). Thus, within close proximity were found materials tending to yield very violent explosions along with others whose reactions could barely be detected. Indeed, some of the violent reactors were actually shown to be slightly less sensitive than some of the relatively inert materials. With these findings, it became apparent that impact sensitivity alone could give a false measure of the potential hazard of a material.

In a broad sense, it appears that the degree of hazard of materials must be defined by two parameters: impact sensitivity and reaction intensity. A method was developed to determine the reaction intensity of impact-induced reactions by measuring the maximum peak pressure of the shock waves generated by the reactions. The technique was found to be practical. The test repeatability was good, particularly in relation to the reaction intensity levels of the various materials of interest.

In the case of solids, it was found that both impact sensitivity and reaction intensity are required to provide meaningful information. In the case of liquids and greases, it was found that the impact sensitivities vary but little for a wide variety of such materials of interest. Accordingly, for such liquids and greases, reaction intensity alone appears to provide the necessary meaningful information.

With the above in mind, equipment and procedures were refined and standardized into two separate test methods: impact sensitivity and reaction intensity. Using the reaction intensity test on a wide variety of liquids and greases, it has been found that all materials tested separate neatly and conclusively into two categories: relatively inert and explosively reactive, with the separation nearly always being achieved in no more than 20 test drops. It appears that the potential hazard of a liquid or grease is not necessarily affected by the presence of trace contaminants. Further, it appears that most

materials presently categorized as having batch-to-batch variance of sensitivity are potentially all hazardous, with the occasional reactions observed in the present sensitivity tests being a sporadic indication of this. The batches of such materials that pass present test methods probably do so because of chance. In a similar connection, the new test method has identified the explosive potential of several other materials which the current test methods falsely show to be nonreactive.

For solids, only one material, Teflon, showed a low reaction intensity rating. On the other hand, several materials, such as Mylar and Kapton, showed reaction intensities not very much higher than Teflon, with the indication being that reactions with these materials were self-quenching in nature. At the same time, sensitivity tests showed that, in general, sensitivity decreases (the 50-percent point goes up) with thinner samples for all materials tested and that sensitivity is significantly influenced by other parameters in addition to the impact energy delivered. Also, Mylar is reported to have had a long, successful service history in certain usages in spite of the fact that it can react in a limited fashion. Weighing these various considerations against the fact that other physical properties make some of the slightly reactive materials preferable to the more inert types in certain service usages, it is concluded that the slightly reactive types need not necessarily be barred from all service. Indeed, it is felt that a more exact definition of the service parameters to which Mylar was exposed, together with reaction intensity and sensitivity information from the two proposed test methods, might provide a most valuable correlation between test and service conditions. Obviously, such a correlation would allow considerably more latitude in the selection of solid materials for specific applications.

2. RECOMMENDATIONS

The two test methods, namely, an impact sensitivity test method and a reaction intensity test method, as outlined in the Appendix of this report, are recommended for serious consideration to replace the current test methods in this area. It is further recommended that an adequate interlaboratory program be initiated to establish beyond a reasonable doubt the repeatability and reproducibility of the two test methods.

All liquids and greases currently considered safe for liquid oxygen service by virtue of having passed any one of the existing impact sensitivity tests should be promptly evaluated by the reaction intensity test. This is recommended in view of the several explosive liquids and greases which the current test methods can sometimes misleadingly rate as nonreactive.

Finally, it is recommended that every effort be made to obtain valid information on the service experience of the materials that have been used and the attendant service conditions. From such information, it is hoped, may be derived the much needed correlation between service and laboratory testing.

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APPENDIX

RECOMMENDED METHODS OF TEST FOR COMPATIBILITY OF MATERIALS WITH LIQUID OXYGEN UNDER IMPACT

1. Scope and purpose. This bulletin covers the determination of the relative sensitivity and potential hazard of materials under impact in the presence of liquid oxygen, using the AFAPL impact tester.

2. Summary of methods. A sample of the test material is placed in a specimen cup, precooled and covered with the liquid oxygen, and placed in the cup holder located in the anvil region assembly of the impact tester. A precooled striker pin is then placed on the sample, using the striker pin guide for centering. The plummet is then dropped from a selected height onto the pin in an effort to produce a controlled level of ignition stimulus in the test sample. Two test methods are included. One of these, the impact sensitivity test, provides a relative measure of the sensitivity of materials with liquid oxygen under impact. The other, the reaction intensity test, provides a relative measure of the potential hazard of materials due to the impact-induced reactions. In the impact sensitivity test, twenty-five drops are made from varied heights in accordance with an "up-and-down" technique, and the drop height corresponding to a 50-percent probability of reaction is determined. In the reaction intensity test, twenty drops are made from a fixed height, and the peak pressures of the shock waves generated in the air by reactions are measured.

3. Test equipment. Complete sets of drawings for the AFAPL impact tester, which consists of an ABMA-type drop-weight apparatus, anvil region assembly, striker pins, specimen cups and sleeves, reaction intensity measuring system, rebound catcher, and other auxiliary equipment, will be furnished upon written request to: Air Force Aero Propulsion Laboratory, Attention APFL, Wright-Patterson Air Force Base, Ohio 45433.

3.1 ABMA-type drop-weight apparatus. The ABMA-type drop-weight apparatus (Fig. 30) consists of three guide rails capable of maintaining vertical alignment under repeated shock conditions, a 20 ± 0.05 -lb plummet weight, a fourth rail which supports an electromagnet for holding or releasing the plummet, a solenoid-operated safety catch to support the plummet when the magnet is not energized, and a base plate.

3.2 Foundation. The foundation for the impact tester is a rigid 2 by 2 by 2-ft (minimum) reinforced concrete block conforming to ASTM Specification C 145 for Solid Load-Bearing Concrete Masonry Units. Four stainless steel foundation bolts protruding from the concrete block are used to fasten the tester base plate to the block.

3.3 Anvil region assembly. The anvil region assembly (Fig. 31) consists of an anvil plate, a specimen cup holder, and a striker pin guide.

3.4 Striker pins and specimen cups. The stainless steel striker pins (Fig. 32) and stainless steel specimen cups (Fig. 33) may be reused (cleaned

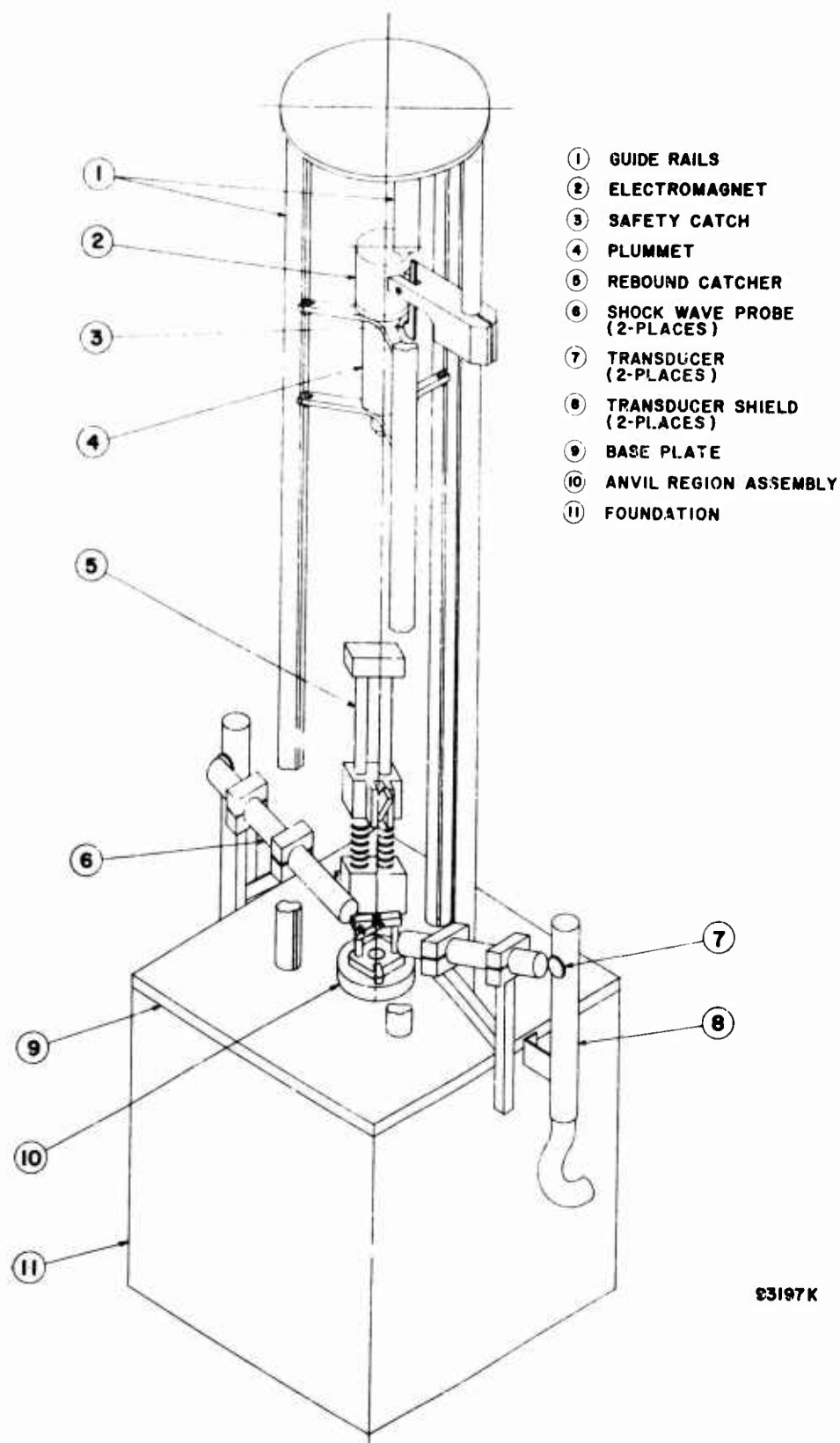


FIGURE 30. AFAPL IMPACT TESTER

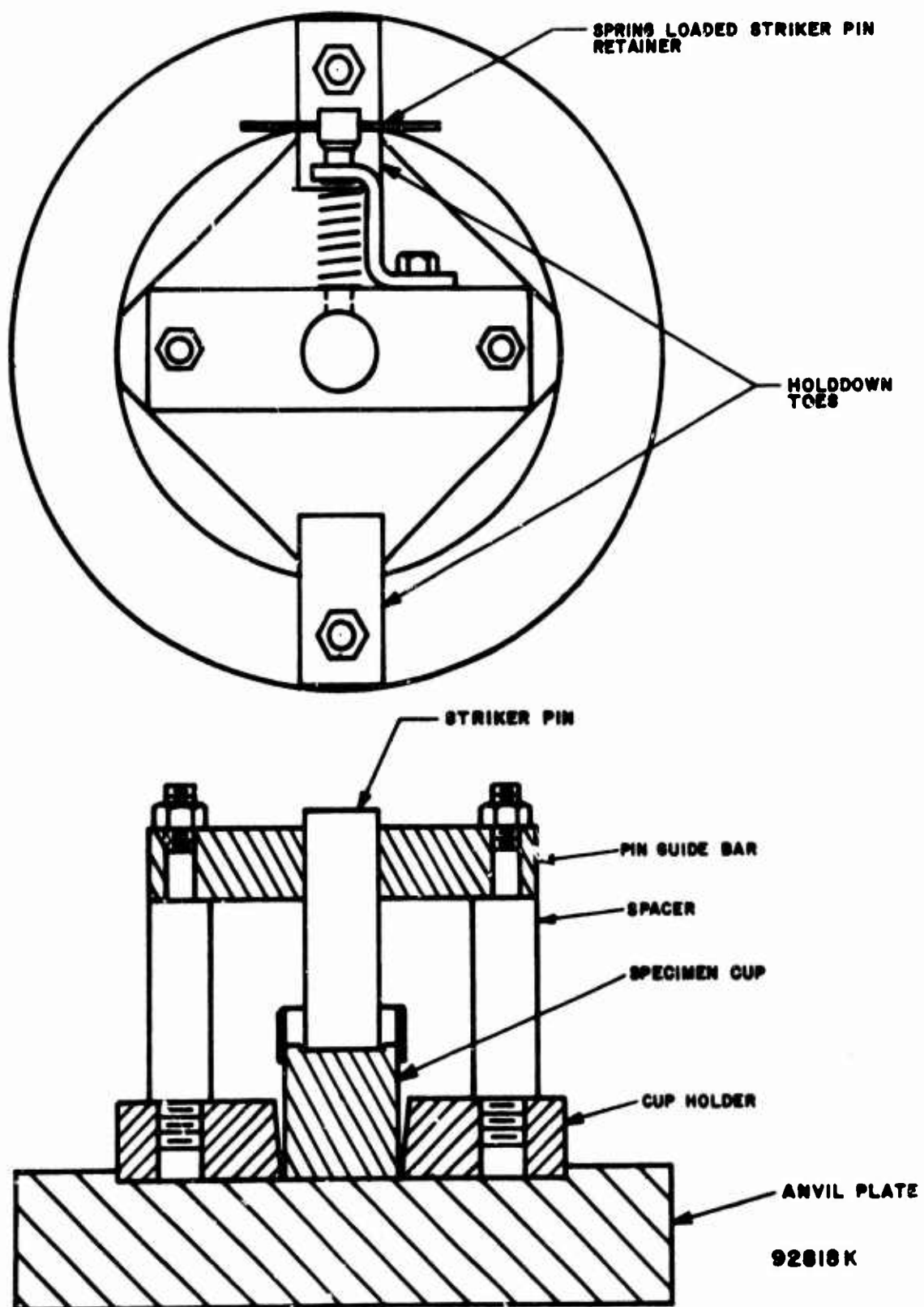
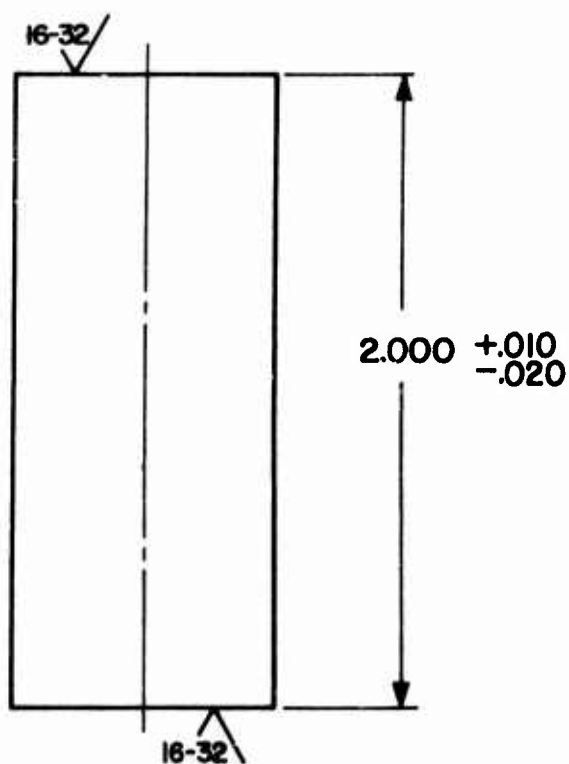
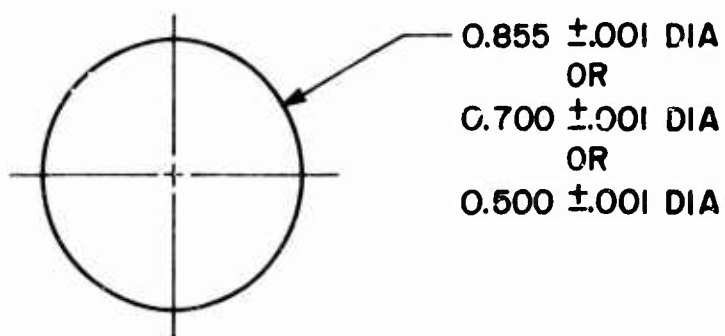


FIGURE 31. ANVIL REGION ASSEMBLY

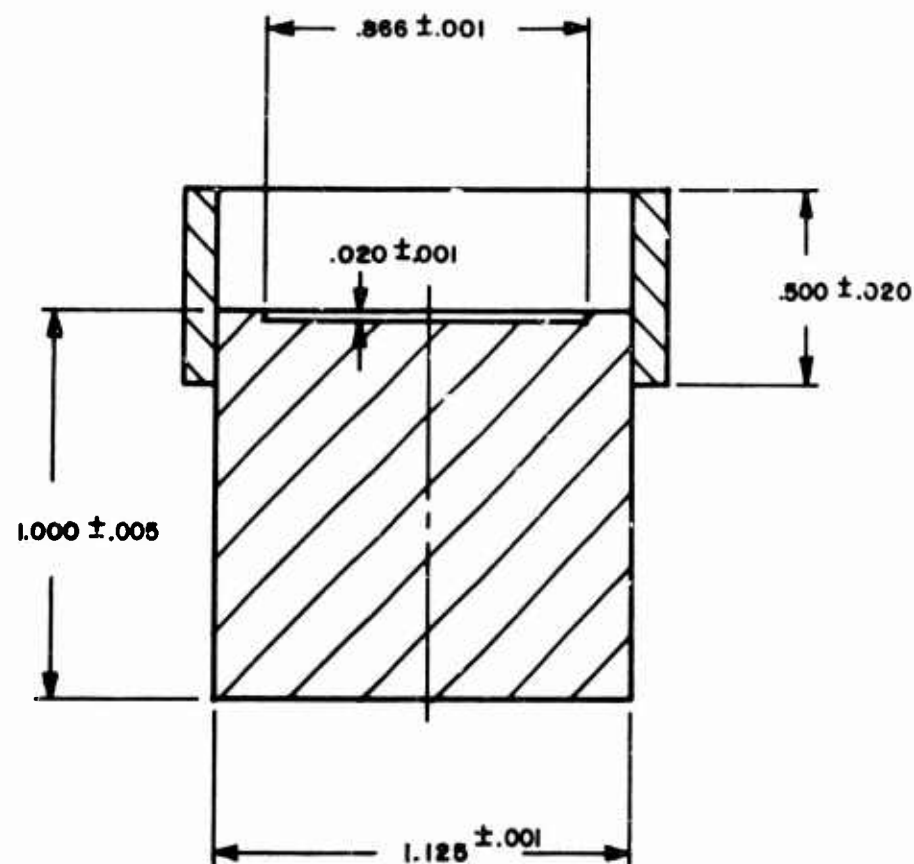
✓ SURFACES MUST BE PARALLEL, FLAT, SQUARE AND TRUE TO EACH OTHER WITHIN .0010 T.I.R.

THE SURFACE FINISH MARKS SHOULD LAY PARALLEL TO EACH OTHER



MATERIAL:
17-4 PH STAINLESS STEEL
HEAT TREAT :
CONDITION H-900

FIGURE 32. STRIKER PIN



SPECIMEN CUP:

MATERIAL:

17-4 PH STAINLESS STEEL

HEAT TREAT:

CONDITION H-900

SLEEVE:

MATERIAL:

CUT FROM 6061-T6 ALUMINUM

TUBING 1.250 O.D. X 0.065 WALL

FIGURE 33. SPECIMEN CUP

and refinished, if necessary) until their dimensions no longer meet the drawing specifications.

3.5 Specimen cup sleeves. The aluminum specimen cup sleeves (Fig. 33), which are pressed into position, are used only once and then discarded.

3.6 Reaction intensity measuring systems. Two identical but independent reaction intensity measuring systems (Figs. 30 and 34) are employed for obtaining peak pressure measurements in the air shock waves produced by reactions. Each system consists of a shock wave probe which serves to capture a portion of the shock wave and guide it essentially unchanged to a remote point of measurement, a piezoelectric transducer, a low-noise transducer cable, a charge amplifier, and a peak pressure meter for obtaining a voltage reading corresponding to the peak pressure of the shock wave. The use of two measuring systems, which are located directly opposite each other, allows a more accurate measurement of directional reactions. The use of the shock wave probes allows the transducers to be shielded from the extraneous effects of violent reactions.

3.6.1 Shock wave probes. Each shock wave probe consists of a stainless steel tube 16 in. long and 1.75 in. in inside diameter.

3.6.2 Pressure Transducers. Each of the two transducers shall be a Kistler Model 506 L or equivalent.

3.6.3 Low-noise cables. Use Kistler Model 131 M or equivalent.

3.6.4 Charge amplifiers. Use Kistler Model 504 or equivalent. On the OUTPUT side of the charge amplifier, use a 50-kc cutoff filter (Kistler Model 544A50 or equivalent).

3.6.5 Peak pressure meters. The peak pressure meter allows rapid reading of the shock wave peak pressure. The circuit diagram for this instrument is shown in Figure 35.

3.6.6 Protection of measuring system. It is essential that the electronic portion of the measuring system located in the test cell (the transducers and cables) be fully protected from the effects of violent reactions. Such protection is afforded as follows:

- (1) The transducer shall be mounted with rubber O-rings to reduce the influence of vibrations from the impacting plummet. The entire transducer, except for the diaphragm, shall be enclosed within the mounting device so that the air shock waves can reach nothing but the diaphragm.

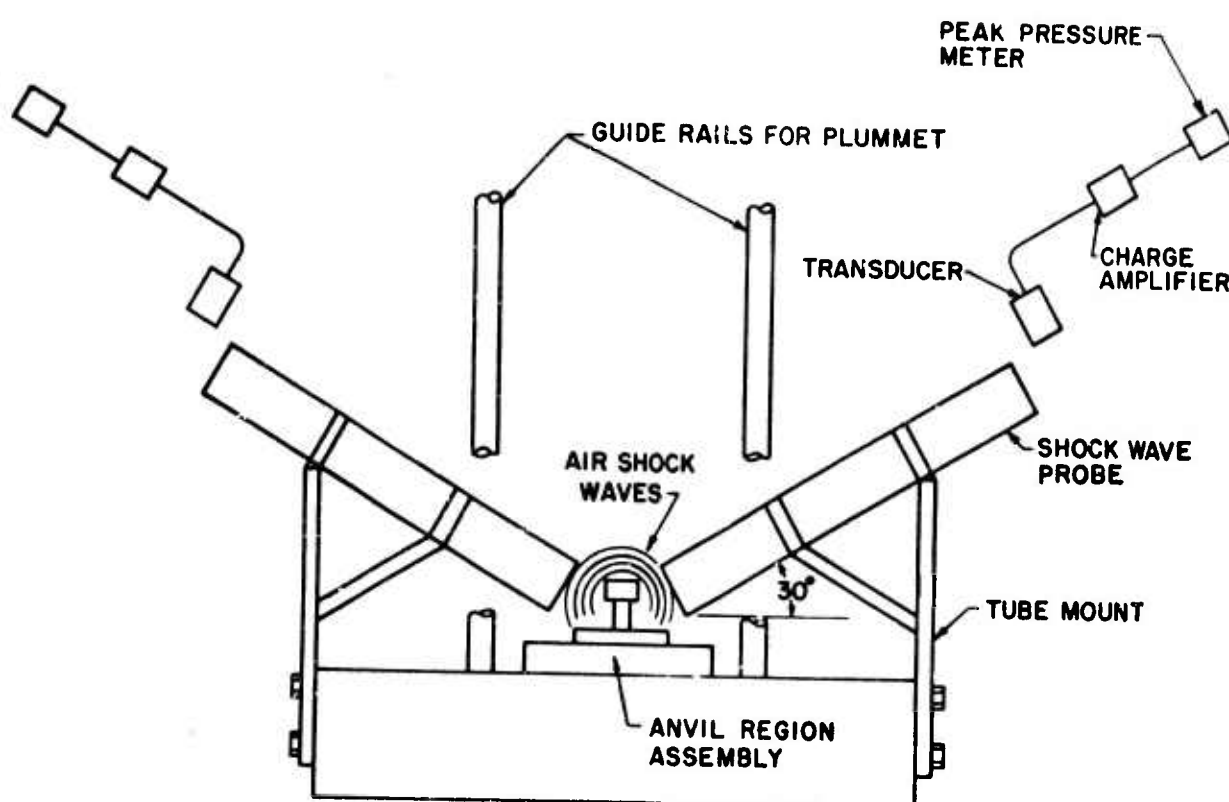


FIGURE 34. REACTION INTENSITY MEASURING SYSTEM

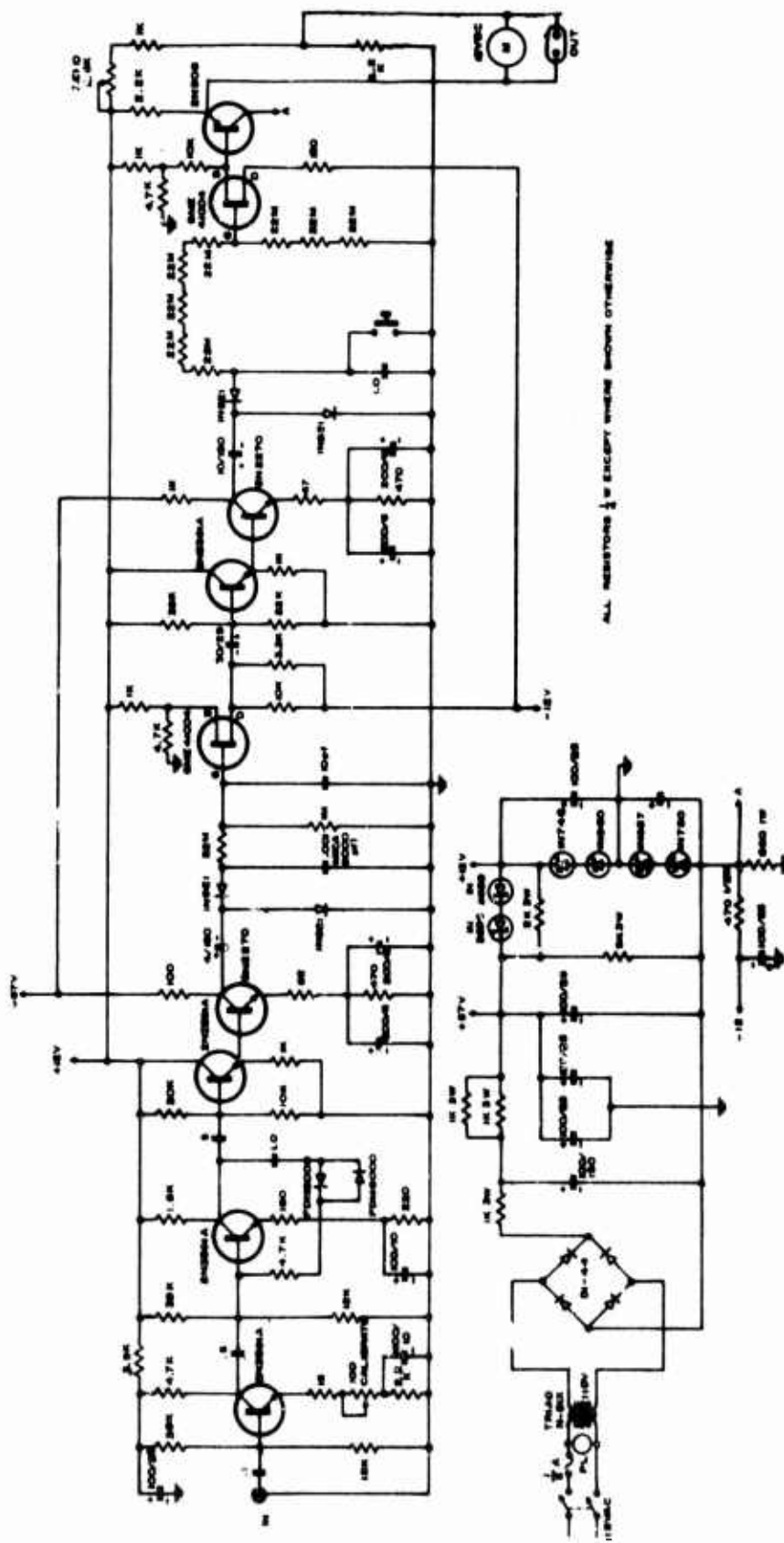


FIGURE 35. CIRCUIT DIAGRAM FOR PEAK PRESSURE METER

- (2) The low-noise cable shall be enclosed in a rigid metal or PVC tubing since any movement of the cable due to air shock waves or flying particles will produce extraneous signals.

3.7 Rebound catcher. The rebound catcher (Fig. 30) catches the plummet after its initial impact since the allowance of secondary impacts would reduce the control of ignition stimuli.

3.8 Test cell. The impact tester shall be housed in a test cell containing a concrete floor. Walls shall be constructed of reinforced concrete or metal to provide protection from explosion or fire hazards. The cell shall be provided with a shatterproof observation window and shall be darkened sufficiently to permit observation of flashes. (The operator shall be located in a darkened area outside the test cell.) Continuous ventilation shall provide fresh air to the test cell. Construction of the cell shall be such that it can be kept thoroughly clean.

3.9 Freezing tray. The freezing tray serves as a container for the pre-cooling of striker pins and specimen cups containing the test samples. A stainless steel tray with a cover, similar to a hospital surgical tray, may be used. The design and dimensions are not critical, except that it must be about 2 in. deep and large enough to hold about thirty pins and thirty cups with room to allow easy handling of the pins and cups by means of forceps. If desired, two smaller freezing trays may be used, one for the pins and one for the cups. The use of asbestos insulation under and around the sides of the tray or trays will retard liquid oxygen boiloff. The tray or trays shall be placed on a table in the test cell in the vicinity of the impact tester such that their lower surface is level.

3.10 Auxiliary equipment. The auxiliary equipment shall consist of stainless steel forceps for handling the specimen cups and striker pins, stainless steel spatulas, liquid oxygen handling equipment such as stainless steel dewar flasks, liquid oxygen protective gloves, lintless laboratory coat, eye protection equipment, and liquid oxygen storage containers. Special handling equipment shall include specimen cup trays, covered storage containers for specimen cups and striker pins, and a vapor-phase degreaser. The following items are also recommended: microburette (10 ml), control panel containing all control devices and measuring instruments, stereomicroscope, micrometer depth gage with leveling blocks, press punch cutter for preparation of plastic specimens, oven, and refrigerator. For checking surface roughness of striker pins and specimen cups, a set of visual roughness comparison standards* or a surface roughness measuring instrument shall be required. Timing instrumentation shall be required to measure the drop time of the plummet to the nearest 1/10 ms. A suitable timing circuit is illustrated in Figure 36.

*American Standard Association, B46.1 - 1962. Surface Texture Standards may be used.

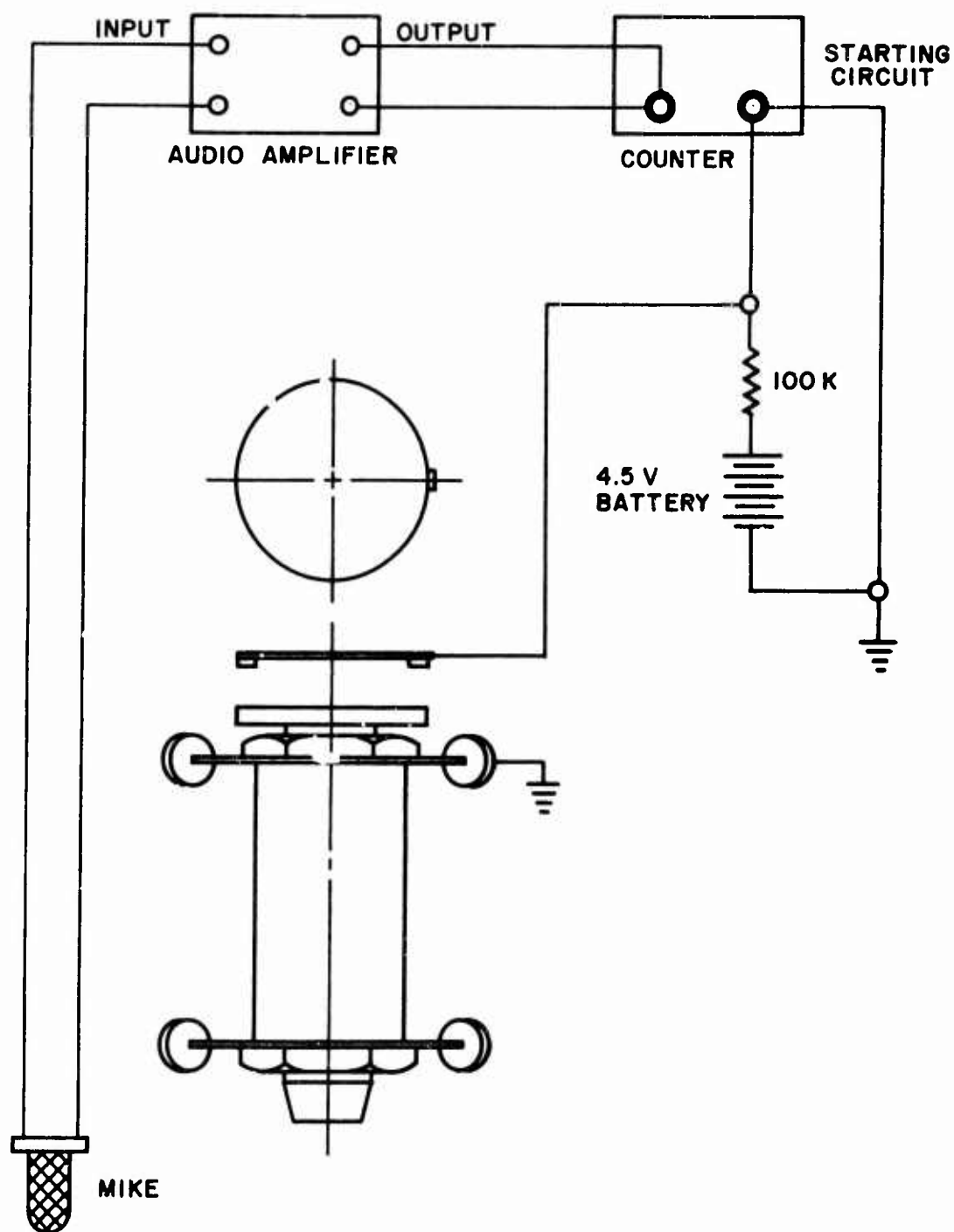


FIGURE 36. FREE FALL TIMING CIRCUIT

4. Materials

- 4.1 Liquid oxygen. The liquid oxygen shall conform to MIL-O-25508.
- 4.2 Trichloroethylene. The trichloroethylene shall conform to MIL-T-27602. Trichlorotrifluoroethane conforming to MIL-C-81302 may be used as an alternative.
- 4.3 Detergent. Use household detergent such as Tide or equivalent.
- 4.4 Sulfuric acid dichromate. Mix 1 liter of concentrated sulfuric acid with 35-ml of saturated sodium dichromate solution.
- 4.5 Steel wool. Grade 00 steel wool is recommended for use.
- 4.6 Abrasive paper. Use 240-grit silicon carbide waterproof abrasive paper.
- 4.7 Miscellaneous materials. Other materials which are required include shop towels, lintless tissue, and distilled water.

5. Safety precautions

- 5.1 Liquid oxygen. The hazards involved with liquid oxygen are very serious. Contact with the skin can cause frostbites resembling burns. Contact with hydrocarbons or other fuels causes an explosion hazard, as such mixtures are usually shock, impact, and vibration sensitive.
- 5.2 First aid. The first-aid procedure for liquid oxygen contact is to flush the affected area with water. This treatment should be followed by medical attention. A safety shower must be available in the immediate area.
- 5.3 Safety rules. The following safety rules must be observed: personnel working with liquid oxygen must be familiar with its nature and characteristics. Approved goggles or face shields, protective clothing, gloves, and boots must be worn during handling or transfer. Such operations shall be performed by not less than two persons as a group. Extreme caution shall be exercised in preventing contact with oils or other combustible materials. All tools must be degreased before use. Precaution shall be taken to prevent accumulation of moisture in lines, valves, traps, etc., in order to avert freezing and plugging with subsequent pressure ruptures. Care shall also be taken to prevent entrapment of liquid oxygen in unvented sections of any system.
- 5.4 Safety equipment. Safety shower and other protective equipment shall be inspected periodically and prior to each handling of liquid oxygen. Personnel leaving the working or storage area shall take steps to make sure that

no oxygen remains absorbed in clothing before smoking or approaching any source of ignition.

5.5 Ventilation. The threshold limit value, that is, the time-weighted average concentration of trichloroethylene believed safe for continuous exposure during a normal 8-hr workday, has been established by the American Conference of Governmental Industrial Hygienists at 100 ppm. Operations employing trichloroethylene should always be conducted in a well-ventilated area. The comparable figure for trichlorotrifluoroethane is 1000 ppm, and normal ventilation is usually adequate. When a ventilation system is employed, an effort should be made to have the natural air currents in the vicinity assist rather than oppose the mechanical ventilation. For cleaning by vapor degreasing, the vapor level shall be controlled by heat input and cooling coils which establish a vapor "ceiling." Such vapor-degreasing units should always be installed in a location that is free from draft conditions and should be ventilated by horizontal slot exhausts.

6. Cleaning and storage of equipment

6.1 General. The impact tester, its accessories, and the test cell shall be kept clean. The guide rails, plummet, anvil plate, striker pin guide, specimen cup holder, shock wave probes, transducer mounts, and base plate of the impact tester shall be cleaned thoroughly at the start of each test and between tests of different materials by using clean shop towels and/or steel wool, and rinsing with fresh trichloroethylene. In addition, the anvil plate, specimen cup holder, striker pin guide, and plummet nose shall be similarly cleaned at least after every tenth test drop. After completion of testing for the day, the impact tester, handling equipment (dewar flasks and forceps), striker pins and specimen cups used in testing, and sample preparation equipment shall be rinsed with fresh trichloroethylene. Cleanliness must be maintained throughout any series of impact testing to minimize the possibility of erroneous test results.

6.2 Test cell. The ceiling and any fixtures in the test cell that could accumulate dust shall be cleaned at least weekly. The concrete floor shall be vacuumed at least weekly and scrubbed when necessary. The air intake shall be filtered and the filter changed at least every 3 months.

6.3 Striker pins. Striker pins shall be cleaned and refinished as follows:

- (1) Wipe with clean shop towels and rinse with fresh trichloroethylene.
- (2) Place a piece of 240-grit abrasive paper on a flat, smooth surface, such as plate glass. Refinish the impacting surface of the pin using a back-and-forth sanding motion to produce a surface finish as specified in Figure 33. During this sanding

procedure, care should be taken to keep the pin axis as close to vertical to the paper surface as possible.

- (3) Pins with scratches or pits too deep to be sanded out should be resurfaced on a lathe and then refinished as in step (2).
- (4) Wash in detergent, rinse with distilled water, and air- or oven-dry.
- (5) Vapor degrease with fresh trichloroethylene for 5 min. Handle pins with clean forceps.
- (6) Store in a clean, covered container. If stored for more than 1 day, rinse with fresh trichloroethylene and air-dry before using. Handle pins with clean forceps.

6.4 Specimen cups. Specimen cups shall be cleaned and refinished as follows:

- (1) Wipe with clean shop towels and rinse with fresh trichloroethylene.
- (2) Using a lathe or similar device to spin the cups, sand the bottom of the specimen depression using 240-grit abrasive paper.
- (3) Cups with scratches or pits too deep to be sanded out should be resurfaced on a lathe and then refinished as in step (2).
- (4) Any cup on which more than a minimum of refinishing and/or resurfacing has been necessary should be checked to see if the depth of the specimen depression has exceeded 0.020 ± 0.002 inch. If so, the cup lip may be machined off to bring the specimen depression depth within tolerance.
- (5) Wash in detergent, rinse with distilled water, and air- or oven-dry.
- (6) Vapor degrease with fresh trichloroethylene for 5 min. Handle cups with clean forceps.
- (7) Store in a clean, covered container. If stored for more than 1 day, rinse with fresh trichloroethylene and air-dry before using. Handle cups with clean forceps.

6.5 Specimen cup sleeves. Specimen cup sleeves shall be cleaned as follows:

- (1) Wash in detergent, rinse with distilled water, and air- or oven-dry.
- (2) Vapor degrease with fresh trichloroethylene for 5 min. Handle sleeves with clean forceps.
- (3) Store in a clean, covered container. If stored for more than 1 day, rinse with fresh trichloroethylene and air-dry before using. Handle sleeves with clean forceps.
- (4) Sleeves are used only once and must be discarded after a drop.

6.6 Electronic equipment. The manufacturers' recommendations should be followed in maintaining electronic equipment such as transducers, low-noise transducer cables, charge amplifiers, and peak pressure meters. Experience has shown that even such things as a small amount of dust or oil in the cable connector can cause erroneous readings.

6.7 Other equipment

6.7.1 Stainless steel ware. Stainless steel ware, such as striker pin holders, forceps, spatulas, and specimen cup trays, shall be cleaned in the same manner as the striker pins (see 6.3). Once integrated into the handling procedure, a thorough rinse of the stainless steel ware with fresh trichloroethylene is the only cleaning necessary.

6.7.2 Glassware. Any glassware, such as microburette, beakers, and pipette, shall be cleaned as follows: If an ordinary detergent wash proves insufficient, soak approximately 2 hr (depending on degree of contamination) in warm sulfuric acid-dichromate solution, rinse with distilled water, and air- or oven-dry.

6.7.3 Liquid oxygen handling equipment. Liquid oxygen handling equipment, such as gaseous oxygen lines (tubing) and liquid oxygen storage containers shall be cleaned thoroughly at least every 3 months with fresh trichloroethylene. Safety manuals on liquid oxygen handling and cleaning procedures shall be followed.

7. Test procedures

7.1 Preparation of test samples

7.1.1 Number of samples. The number of samples to be prepared for a test is dependent both on the tendency of the material to "float" during

precooling (see 7.2) and on the test procedure to be used. The impact sensitivity test requires twenty-five samples, and the reaction intensity test requires twenty samples. In either case, experience will show how many extra samples to prepare.

7.1.2 Liquid (Oils). Liquid samples shall be prepared as follows: Shake liquid well before use, and allow air bubbles to float out of solution. Use a clean 10-ml microburette in handling the liquids. For viscous materials, a microsyringe may be used. Determine by trial and error technique the volume of liquid required to give a sample thickness of 0.020 in. near the center of a cup. Then, after affixing the specimen cup sleeves to the test cups as in 7.1.5, use the determined volume to fill the test cups. A micrometer gage with leveling blocks is suggested for measurement, and the work table should be level. Proceed as specified in 7.2.

7.1.3 Semisolids (greases). Grease samples should be prepared as follows: Press sufficient sample material (a slight excess) in the specimen cup with the clean stainless steel spatula to form a uniform sample free of air bubbles and void spots. Scrape off the excess sample grease with the spatula until a smooth surface level with the edge of the specimen cup is achieved. Remove excess sample from the outer perimeter of the specimen cup with clean lintless tissue, and affix sleeves as in 7.1.5.

7.1.4 Solids. Samples of solids from the application products should be cut and prepared as follows: The diameter of the samples should be 0.860 ± 0.003 inch. The thickness should be that of the application product if at all possible, with the limiting factor being the greatest thickness (approximately 0.30 in.) that can be submerged in liquid oxygen with the sleeve and striker pin in place. Otherwise, samples of the above diameter and having a thickness of 0.020 in. should be specially prepared from the application product, simulating as closely as possible the surface finish of the application product on both sides. Measure the thickness of each sample and record same. Clean samples by thoroughly rinsing in fresh trichloroethylene and air- or oven-dry, and, hereafter, handle samples with clean forceps. If trichloroethylene produces adverse effects, clean with a detergent wash, rinse with distilled water, and air- or oven-dry. Place in clean specimen cup with sleeve (see 7.1.5) and proceed in accordance with 7.2.

7.1.5 Affixing of specimen cup sleeves. Use clean sleeve pressing tool and small clean bench press to press the specimen cup sleeves onto the specimen cups. Handle cups and sleeves with clean forceps.

7.2 Precooling

7.2.1 Definition. The term precooling as used herein means to lower the temperature of the material being cooled to the boiling point of liquid oxygen at one atmospheric pressure (-297°F). Throughout the precooling procedure,

the specimen cups containing the test sample and the striker pins shall be handled with clean forceps.

7.2.2 Samples. Samples shall be precooled as follows: Proceed with extreme care to minimize cracking of the sample in the cup. Place specimen cups (with sleeves) containing the test sample in a clean, levelled freezing tray. Slowly pour enough liquid oxygen into the tray to cover just the bottom, taking care not to splash liquid oxygen onto the warm test sample. After approximately 2 min, a considerable reduction in the rate of boiling will indicate that the tray surfaces have been precooled. Then, slowly add liquid oxygen into the tray to a level about one-half the total height of the specimen cup and sleeve, again taking care not to splash liquid oxygen onto the sample. Keep the liquid oxygen at about this level until all samples are completely frozen. Then, slowly add sufficient liquid oxygen to overflow into the specimen cups. Do not pour liquid oxygen directly into the cups even after the samples have frozen as this will increase the probability of sample breakup. Make a visual check to assure that the sample does not contain a void or hole and that the material has not separated from the bottom of the cup and floated. Discard any sample that does not pass this inspection. Samples need not be discarded for cracking as long as pieces do not separate and float. In the case of solid samples, floating need not be of concern as they will be weighted down to the bottom of the cups by the pins. In all cases, perform drop tests within 4 hr after precooling, keeping cups completely submerged and the tray cover on until test is complete.

7.2.3 Striker pins. A second freezing tray may be used to precool the striker pins. Add sufficient liquid oxygen to the tray containing the striker pins to submerge the pins completely. Do not use the pins until the vigorous boiling of the liquid oxygen ceases. Keep the pins submerged and the tray cover on until the test is complete.

7.3 Test drops. The following procedure shall be used in making test drops:

- (1) Adjust the magnet on the fourth rail of the impact tester to the desired height.
- (2) Arm the trigger on the rebound catcher.
- (3) Transfer a precooled specimen cup from the freezing tray to the specimen cup holder by means of clean forceps, taking care not to spill the liquid oxygen in the cup.
- (4) Place a precooled striker pin in the striker pin guide and lower it gently onto the sample surface by means of clean forceps. No "topping" with liquid oxygen should be necessary if the test drop is made within 20 sec.

- (5) Leave the test cell, close the door, and turn off the test cell light.
- (6) Release the plummet safety catch.
- (7) Release the plummet.

7.4 Blank drops

7.4.1 General. Blank drops shall be performed in accordance with the procedure set forth in 7.3 except that no sample material shall be placed in the specimen cups. Blank drops are used primarily to check the drop time of the plummet, a measurement which indicates whether the tester is in good working order. Such drops shall be made whenever a new tester is installed, after a tester has been overhauled, at intervals of not more than 1 month, whenever there is reason to suspect the results, or as required in the test sequence. In all such cases, a minimum of three drops shall be made from a height of 43.3 inches.

7.4.2 Maximum allowable drop time. The maximum allowable drop time is 3 percent more than the theoretical time for free fall as determined using the prevailing gravitational acceleration. This value shall be calculated to the nearest 1/10 millisecond.

7.4.3 Drop time check. Each time a blank drop is made, the drop time of the plummet shall be measured to the nearest 1/10 millisecond. If this measured value equals or exceeds the maximum allowable drop time, the tester and/or the free fall timing circuit is in need of immediate attention.

7.5 Impact sensitivity test

7.5.1 General. The impact sensitivity test is designed to yield a statistically reliable estimate of the drop height at which the probability of reaction of the test sample is 50 percent. The drop height so determined is called the 50-percent point and may be used as a relative sensitivity rating for the test sample. The test is comprised of twenty-five test drops made from varied drop heights in an up-and-down fashion.

7.5.2 Data sheet. Use the data sheet shown in Table 16. In this data sheet, the drop heights are selected to the nearest 1/10 in. such that their logarithms are spaced at intervals of 0.10. This logarithmic scale is required for the statistical computation of the 50-percent point.

7.5.3 Striker pins. Any one of the three striker pin diameters shown in Figure 32 may be used in the impact sensitivity test. For liquids and greases, the 0.855-in. diameter striker pin shall be used, along with a standard sample thickness of 0.020 inch. For solids, the choice of the pin

TABLE 16. DATA SHEET FOR IMPACT SENSITIVITY TEST

Sample _____ Date _____ Operator _____
 Temperature _____ Barometric Pressure _____ Humidity _____
 Sample Thickness _____ Striker Pin Dia. _____

Maximum allowable drop time from 43.3 in.: _____ msec

Measured drop time from 43.3 in.: Before 1st Test Drop _____ msec

After 10th Test Drop _____ msec

After 20th Test Drop _____ msec

Test Drop No.

1955 Drop Test																											
Drop Ht.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	X's	O's
48.0																											
38.1																											
30.3																											
24.0																											
19.1																											
15.2																											
12.0																											
9.6																											
7.6																											
Total																											

Drop Ht.	i	N _i	i N _i
	5		
	4		
	3		
	2		
	1		
	0		

N = ____ A = ____

50-percent Point:

$$\bar{X} = \log (\text{lowest drop ht.}) + 0.1 \left(\frac{A}{N} \pm \frac{1}{2} \right)$$

=

Antilog \bar{X} = _____ in.

diameter depends upon the sample material and sample thickness. In many cases, it may be desirable to conduct separate tests, each using one of the three different pin diameters, preferably with a sample thickness equal to that of the application product (see 7.1.4).

7.5.4 Reaction intensity measurements. Use of the reaction intensity measuring system is not required in the impact sensitivity test.

7.5.5 Evidence of reaction. On each test drop, the operator shall watch for any flash or burning and listen for any sound or reaction. Immediately after the test drop, he shall inspect the specimen cup and striker pin for other evidence of reaction, such as discolored residue, pits or radially expanding erosions of the metal surfaces, or radially expanding patterns in the sample remains. If any such evidence is found, no matter how insignificant it may seem, the operator shall decide that a reaction has occurred.

7.5.6 Laboratory certification. A 50-percent point obtained by this test method may not be considered comparable to results obtained by another organization unless the laboratory has been "certified" in accordance with paragraph 7.5.6.1.

7.5.6.1 Certification procedure. A laboratory may become certified by obtaining 50-percent points within limits prescribed by the Air Force on control samples specified by the Air Force (see 3.).

7.5.7 Procedure. The following procedure shall be followed in performing the impact sensitivity test.

7.5.7.1 Blank drops. For each test, three blank drops shall be made from a height of 43.3 in. (see 7.4). The first blank drop shall be made immediately before the first test drop, the second after the tenth test drop, and the third after the twentieth test drop. The drop times shall be recorded on the data sheet, together with the maximum allowable drop time. If any of the three drop times equals or exceeds the maximum allowable drop time, the entire test shall be invalidated. No further test drops shall be made until the trouble has been eliminated.

Note: Do not make blank drops using the 0.5-in. diameter striker pins, as these pins will be permanently deformed in drops from 43.3 in. with no sample in the cup. For impact sensitivity tests using these pins, substitute one of the other striker pin diameters shown in Figure 32 for the blank drops only.

7.5.7.2 Test drops. The first test drop shall be made from a height of 19.1 inches. After determining whether a reaction has occurred, (see 7.5.5), proceed in accordance with the following criteria:

- (1) If evidence of a reaction is found, place an "X" in the appropriate box on the data sheet. The second test drop shall then be made from the next lower drop height.
- (2) If no evidence of a reaction is found, place an "O" in the appropriate box on the data sheet. The second test drop shall then be made from the next higher drop height.

The remaining twenty-three test drops shall be made in a similar manner with the choice of the next test drop; height always being based on the result of the immediately preceding test drop.

Note: If the approximate location of the 50-percent point can be anticipated from prior test experience, the first test drop should be made from the drop height nearest the anticipated 50-percent point rather than from 19.1 inches.

7.5.7.3 Computations. For analysis, use the symbol (X or O) with the least number of occurrences. To compute the 50-percent point, use the following procedure together with the inset table provided on the data sheet.

- (1) Enter all drop heights at which the symbol under analysis occurred in the "Drop Ht" column, starting with the lowest drop height at the bottom of the column opposite the number zero in the "i" column. The rest of the drop heights are to be entered in ascending order (do not skip any heights or lines) up through the highest drop height at which the symbol occurred.
- (2) In the " N_i " column, enter the number of occurrences of the symbol at each drop height.
- (3) In the " iN_i " column, enter the product of the number in the "i" column by the adjacent number in the " N_i " column.
- (4) Total the " N_i " and " iN_i " columns, and let the totals be represented by

$$N = \sum N_i$$

$$A = \sum iN_i$$

- (5) Compute the 50-percent point using the following formulas:

$$\bar{X} = \log (50\text{-percent point})$$

$$= \log (\text{lowest drop height}) + 0.1 \left(\frac{A}{N} \pm \frac{1}{2} \right)$$

where the lowest drop height is the lowest drop height at which the symbol under analysis occurred, and:

$+\frac{1}{2}$ is used if the "O's" are under analysis

$-\frac{1}{2}$ is used if the "X's" are under analysis.

The 50-percent point in in. is thus

50-percent point - antilog \bar{X}

7.5.8 Example test. Table 17 presents an example of a completed impact sensitivity test data sheet as an illustration of the manner in which the test and computational procedures are performed.

7.6 Reaction intensity test

7.6.1 General. The reaction intensity test is designed to yield an evaluation of the potential fire or explosive hazard of a material. It consists of three blank drops and twenty test drops, all made from a height of 43.3 in. using the 0.855-in. diameter striker pin only. On each test drop, two simultaneous measurements are made of the peak pressure of the air shock wave emanating from the area of the specimen cup. In addition, notations are made if flashes, burning, or postdrop evidences of reaction propagation are observed. This test procedure assumes the use of the instrument system described in paragraph 3.6.

7.6.2 Data sheet. Use the data sheet shown in Table 18.

7.6.3 Transducer location check. Prior to the conduction of the test, check to see that the transducers and shock wave probes are located as specified, as deviations from the specified locations will cause deviations in the pressure measurements.

7.6.4 Instrument settings. In using this test procedure, peak pressure readings ranging from less than 0.20 psi to greater than 150 psi may be encountered, depending on the material being tested. Since the pressure measuring system is not capable of providing accurate readings over the entire range with one setting, it will be necessary to select the range of primary interest. Table 19 gives the range of accurate readings for each of several settings of the charge amplifier. In testing new materials on which there is no prior knowledge, it may be necessary to perform extra test drops to find the desired range.

7.6.5 Laboratory certification. Peak pressures yielded by this test may not be considered comparable to results obtained by another organization unless the laboratory has been certified in accordance with paragraph 7.6.5.1.

TABLE 17. EXAMPLE OF COMPLETED DATA SHEET FOR
IMPACT SENSITIVITY TEST

Sample _____ Date 8-29-66 Operator BT
 Temperature 80°F Barometric Pressure 30.00 in. Hg Humidity 50%
 Sample Thickness 0.020 in. Striker Pin Dia. 0.855 in.

Maximum allowable drop time from 43.3 in.: 488.1 msec

Measured drop time from 43.3 in.: Before 1st Test Drop 483.1 msec

After 10th Test Drop 482.7 msec

After 20th Test Drop 481.7 msec

Test Drop No.

Test Drop No.																												
Drop Ht.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	X's	O's	
48.0																												
38.1																												
30.3																												
24.0		X		X		X											X		X					X		6		
19.1	O		O		O		X		X			X		X		O		O		X		O		O		O	5	7
15.2								C		X		O		O		O						O					1	5
12.0											O																1	
9.6																												
7.6																												
Total																									12	13		

Drop Ht.	i	N _i	i N _i
	5		
	4		
	3		
24.0	2	6	12
19.1	1	5	5
15.2	0	1	

N = 12 A = 17

50-percent Point:

$$\bar{X} = \log (\text{lowest drop ht.}) + 0.1 \left(\frac{A}{N} \pm \frac{1}{2} \right)$$

$$= 1.181 + 0.1 \left(\frac{17}{12} - \frac{1}{2} \right)$$

$$= 1.2727$$

Antilog \bar{X} = 18.7 in.

TABLE 18. DATA SHEET FOR REACTION INTENSITY TEST

Sample _____ Date _____ Operator _____

Temperature _____ Barometric Pressure _____ Humidity _____

Sample Thickness _____ Striker Pin Dia. _____ Drop Height _____

Test Drop No.	Flash or Burning	Char Marks	Peak Pressure Meter I, volts	Peak Pressure Meter II, volts	Charge Amplifier, psi/volt	Peak Pressure, psi
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
Total						

Maximum allowable drop time:

_____ msec

Measured drop time:

Before 1st Test Drop _____ msec

After 10th Test Drop _____ msec

After 20th Test Drop _____ msec

Description of Flash,
Burning, or Char Marks

Average Peak Pressure _____ psi

Maximum Peak Pressure _____ psi

TABLE 19. CONVERSION FACTORS FOR PEAK
PRESSURE MEASUREMENTS

Charge Amplifier Range, psi/volt	Conversion Factor	Accuracy Range, (a) psi	
		From	To
10	0.1	0.01	1
20	0.2	0.02	2
50	0.5	0.05	5
100	1.0	0.1	10
200	2.0	0.2	20
500	5.0	0.5	50
1 K	10.0	1.0	100
2 K	20.0	2.0	200

(a) The peak pressure meter provides a nearly constant 100 to 1 amplification of input signals over the range of 1 to 100 mv. Signals of less than 1 mv will simply not register, while signals of more than 100 mv will oversaturate the capacitors, making the resultant readings unreliable.

7.6.5.1 Certification procedure. A laboratory may be considered certified when peak pressures have been obtained within limits prescribed by the Air Force on control samples specified by the Air Force (see 3.).

7.6.6 Procedure. The following procedure shall be followed in performing the reaction intensity test.

7.6.6.1 Instrument settings. Before commencing the test, the instrument settings shall be adjusted as follows:

- (1) On each charge amplifier, dial the charge sensitivity of the corresponding transducer on the TRANSDUCER SENSITIVITY dial, and then lock the dial.
- (2) On each charge amplifier, set the RANGE dial to the desired range (see 7.6.4), turn the power switch ON, turn the TIME CONSTANT switch to SHORT, and depress the GND button briefly. Record the RANGE setting on the data sheet (this need be entered only once).
- (3) On each peak pressure meter, turn the POWER switch ON, depress the RESET button, and set the meter needle to zero using the ZERO knob.
- (4) Monitor the peak pressure meters continuously throughout the test drops. A needle movement that does not immediately follow the impact of the plummet is an erroneous result. If such troubles should persist, the first system check should be for contaminants in the cable connectors. If cleaning all connectors does not rectify the problem, consult the manufacturer's operating manuals.

7.6.6.2 Blank drops. For each test, three blank drops shall be made from a height of 43.3 in. (see 7.4). The first blank drop shall be made immediately before the first test drop, the second after the tenth test drop, and the third after the twentieth test drop. The drop times shall be recorded on the data sheets, together with the maximum allowable drop time. If any of the three drop times equals or exceeds the maximum allowable drop time, the entire test shall be invalidated. No further test drops shall be made until the trouble has been eliminated.

7.6.6.3 Test drops. A total of twenty test drops (see 7.3) shall be made, all from a height of 43.3 inches. During each test drop, watch closely for visible flash or burning of the sample material, checking the appropriate column on the data sheet if such evidence is observed. After each test drop, record the two peak pressure meter readings in the columns provided. Then enter the test cell and inspect the striker pin and specimen cup for char

marks (discolored residue, pitting or radially expanding erosions of the metal surfaces, and/or radially expanding patterns in the sample remains). If any such evidence is found, check the column provided on the data sheet. After leaving the test cell, discharge the peak pressure meters by depressing the RESET button.

7.6.6.4 Description of flash, burning, or char marks. After the twenty test drops are completed, enter in the space provided on the data sheet a brief description of the general nature of flashes, burning, or char marks observed during the test.

7.6.6.5 Computations. For each test drop (exclude blank drops), multiply the higher of the two peak pressure meter voltage readings by the appropriate conversion factor in Table 19. This product is the peak pressure in psi and shall be entered in the right-hand column on the data sheet. The average peak pressure is then computed by dividing the sum of the right-hand column by 20. The maximum peak pressure is simply the highest number in the right-hand column.

7.6.7 Example test. Table 20 presents an example of a completed reaction intensity test data sheet as an illustration of the manner in which the test and computational procedures are performed.

TABLE 20. EXAMPLE OF COMPLETED DATA SHEET FOR
REACTION INTENSITY TEST

Sample _____ Date 2-14-66 Operator BT
Temperature 70°F Barometric Pressure 29.35 in. Hg Humidity 39%
Sample Thickness 0.020 in. Striker Pin Dia. 0.855 in. Drop Height 43.3 in.

Test Drop No.	Flash or Burning	Char Marks	Peak Pressure Meter I, volts	Peak Pressure Meter II, volts	Charge Amplifier, psi/volt	Peak Pressure, psi
1	X	X	2.1	4.4	2K	88
2	X	X	3.7	2.0		74
3	X	X	3.2	5.0		100
4	X	X	2.7	4.8		96
5	X	X	2.0	3.8		76
6		X	0.4	0.7		14
7	X	X	2.1	2.3		46
8	X	X	3.0	4.6		92
9	X	X	2.3	2.1		46
10	X	X	3.1	2.6		62
11	X	X	2.6	3.0		60
12	X	X	4.3	5.3		106
13	X	X	4.9	1.5		98
14	X	X	4.2	5.4		108
15	X	X	5.5	3.1		110
16	X	X	2.3	2.0		46
17	X	X	4.2	5.1		102
18	X	X	3.3	2.7		66
19	X	X	4.6	5.7		114
20	X	X	3.0	2.1		60
Total						1564

Maximum allowable drop time:

488.1 msec

Measured drop time:

Before 1st Test Drop 482.6 msec

After 10th Test Drop 481.9 msec

After 20th Test Drop 483.0 msec

Description of Flash,
Burning, or Char Marks

Flashes were large and bright. Char marks consisted of small amounts of brown residue, some pitting, and large flow marks.

Average Peak Pressure 78 psi

Maximum Peak Pressure 114 psi

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13. ABSTRACT This report summarizes the work done toward the development of suitable test methods for evaluating the compatibility of materials with liquid oxygen under impact. The work indicates that the potential explosive hazard of a material may be defined by two parameters: impact sensitivity and reaction intensity. Impact sensitivity is measured in terms of the drop height at which the material has a 50-percent probability of reaction. Reaction intensity is measured in terms of the maximum peak pressure of the air shock wave generated by the reaction. Detailed test methods for determining impact sensitivity and reaction intensity are proposed. It is recommended that tests be run on a wide variety of liquids, greases, and solids, in strict accordance with the proposed test methods, in order to establish their repeatability and reproducibility.			

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